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Rheological Studies on Pretreated Feed and Melter Feed from C-104 and AZ-102

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Prepared for CH2M Hill Hanford Group
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Summary

Rheological and physical properties testing was conducted on actual AZ-102 and C-104 melter feed samples prior to the addition of glass formers and secondary waste products. Analyses were repeated on the C-104 samples following the addition of simulated Sr/TRU secondary waste. Analyses were repeated again following addition of glass formers to both AZ-102 and C-104 samples. Samples from both feeds were tested at the target solids concentrations of 5, 15, and 25 wt%[1]. This data on actual waste is required to validate and qualify results obtained with simulants.

The AZ-102 feed was received for this task after washing and leaching. The AZ-102 feed contained 9.54 wt% solids. At this solids concentration, no standing liquid was observed in the AZ-102 feed. The C-104 feed was also received following washing and leaching. The AZ-102 feed contained 20 wt% solids and contained roughly 15 vol% standing liquid.

The initial AZ-102 and C-104 feeds displayed very different rheological properties. The initial 5 and 15 wt% C-104 feeds displayed near Newtonian behavior, and the 25 feed has a small yield stress of ~5 Pa giving it a slight Bingham Plastic component. The viscosity of the feeds at 33 s^{-1} were 9.8, 15, and 160 cP for the 5, 15, and 25 wt% feeds respectively with very little if any thixotropic behavior. By comparison, the AZ-102 feed showed much higher initial viscosities of 12, 530, 900, and 4600 cP for the 5, 15, 20, and 25 wt% feeds. In addition, the AZ-102 feeds displayed significant irreversible shear thinning. Surprisingly, no significant temperature effects were seen for the C-104 or AZ-102 samples measured at 25 and 50°C.

Very little change in rheological properties was observed following secondary waste product and glass former addition to the C-104 feed while the yield and viscosity the AZ-102 feed dropped following glass former and secondary waste addition. Since the addition of dry glass formers to the AZ-102 feed should have increased the total solids load, it would have been expected to see an increase the rheological properties. The observed decrease is most likely the result of the irreversible shear thinning of the AZ-102 samples as the testing progressed.

A mixing and aging study was conducted on the 15 wt% AZ-102 and 25 wt% C-104 feeds following glass former and secondary waste product additions. The additions were made and the slurries were stirred at a rate that delivered the same energy per volume as expected in the River Protection Project Waste Treatment Plant flow sheet. Slurries were stirred at this rate for one week. The rheology of the mixed slurries were examined at 1 hr, 1 day and 1 week. The yield and viscosity of the AZ-102 slurry decreased over the one week mixing period consistent with the previous observations of irreversible shear thinning in this slurry. By contrast the yield and viscosity of the C-104 slurry increased over the one week mixing period. The C-104 slurry yield increased from 28 Pa after one hour to 56 Pa after one week. Over this same period, the viscosity of the C-104 slurry increased from 910 cP to 1700 cP at $33 s^{-1}$.

Following the mixing study, the 15 wt% AZ-102 and 25 wt% C-104 slurries were allowed to settle for one week. The AZ-102 sample contained standing liquid, which was removed, while the C-104 sample contained no standing liquid. The rheology of the settled solids were then examined. The yield and viscosity of the AZ-102 settled solids were higher than similar testing after one week of mixing. This increase was expected and attributed to increased solids loading

[1] In this report, wt% solids are based on residual mass after 24 hours at 105°C.

resulting from removal of the standing liquid. The viscosity of the C-104 settled material was similar to the results after one week of mixing, but the yield increased to approximately 80 Pa.

The results of the mixing and aging studies suggest that while the AZ-102 initial feed and final melter feed material may be very difficult to mix and transport, irreversible shear thinning significantly reduces the yield and viscosity. Therefore, exposing this material to some initial shearing may be an option for reducing these critical transport properties. By contrast, the yield and viscosity of the initial C-104 material did not display significant irreversible shear thinning. The yield and viscosity of the C-104 melter feed increased with mixing and the yield increased further with aging. These results suggest the final C-104 melter feed could be very difficult to mix and transport on a 25 wt% basis.

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1.0 Introduction

The scope of the present work was to obtain physical and rheological data on actual high-level waste melter feed samples. The physical and rheological properties of the high level waste melter feed are important considerations in the selection of the melter feed preparation flowsheet and processing equipment such as mixers, pumps, piping and sampling. Measurements on actual waste are required to validate and qualify results obtained with simulants. Measurements were conducted to the quality requirements in PNNL's Standards-Based Management System (SBMS).

Actual samples from tanks AZ-102 and C-104 were used in this testing. Multiple samples from each tank were received from Hanford's 222-S laboratory. Using this material, a composite was prepared for each of the tanks. Each of these composites were then processed through caustic leaching, washing and dewatering steps as described in Brooks et al. (2000a, 2000b).

Solids concentration, settling rate, density and shear stress versus shear rate were measured on the samples after adjusting the samples to 3 solids concentrations (nominally 5, 15 and 25 wt%)[2]. The solids content was first adjusted by decanting standing liquid and then evaporation under a flow of air as needed for the more concentrated samples. The same measurements were repeated on the C-104 samples following the addition of simulated Sr/TRU secondary waste. Analyses were repeated again following addition of glass formers to both AZ-102 and C-104 samples. The original work scope included testing of the AZ-102 evaporated feed following addition of secondary waste products. However, given the small volume of secondary waste products to be added, this testing was deleted as detailed in the Appendix A to Test Plan 29953-073 contained in Appendix B.

Mixing and aging studies were conducted on the 15 wt% solids AZ-102 and 25 wt% solids C-104 slurries with glass formers and secondary waste products. The slurries were placed in a mixing vessel at a shear rate consistent with that expected in the River Protection Project Waste Treatment Plant. Glass formers and secondary waste products were added and the mixing continued for 1 week. During this week, shear stress versus shear rate analyses were conducted after 1 hour, 1 day and 1 week. The slurries were then left undisturbed for 1 week before a shear stress versus shear rate analysis was conducted on the settled solids.

Following addition of secondary waste products and glass formers, the resulting melter feed slurries had a higher solids content. However, in this report, samples will be referred to according to the target solids content for the evaporated feeds (e.g., the feed evaporated to 25 wt% solids is still referred to as the 25 wt% solids slurry event after the addition of dry glass formers.

This report describes the experimental approach and results of the testing. Specifications for this work were provided to Battelle by BNFL under Task Specification Numbers TS-W375HV-PR00011, and TS-W375HV-PR00012. This report also provides a means of transmitting to BNFL the completed test plan and analytical data[3].

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[2] In this report, wt% solids are based on residual mass after 24 hours at 105°C.
[3] Results presented in this report are based on work conducted under Technical Procedure 29953-010, and Test Plan 29953-73.

2.0 Experimental Approach

2.1 Simulated Secondary Waste Products

Glass formulations provided by Catholic University of America's Vitreous State Laboratory (VSL) required significant quantities of secondary waste products that were not available from the actual waste pretreatment testing. Therefore, AZ-102 testing was conducted with the actual secondary waste products from pretreatment studies on actual waste while testing on C-104 material was conducted with simulated secondary waste products. This section provides details on the synthesis of the simulated secondary waste products.

2.1.1 Cs Eluant Simulant

The Cs eluant simulant composition was generated based on the concentrations of materials as reported in, "Estimation Of Physical Properties Of Tank 241-AN-107 Cesium And Technetium Eluate Concentrate Blend" by Alexander S. Choi, Savannah River Technology Center. In the document's appendix the evaporated Cs eluant speciation is described in terms of moles of component generated per hour. Using this information a simulant spreadsheet was created where the individual components reported (metals, anions, and water) were summed to give the total amount of moles for each of the individual metals, anions, and the total water generated per hour. To compose the simulant, it was assumed initially that the bulk of metals would be introduced as nitrate salts, the total acid concentration would be generated by nitric acid and HCl, and the total chloride would be supplied solely through the addition of HCl. A later check on the total amount of anions introduced by this assumption agreed well with the total amounts present in the spreadsheet. In the actual simulant preparation, the total amounts of each component needed then were scaled so that ca. 100 to 150 mLs of total solution would be prepared (1/40th of the per hour spreadsheet amount). Table 2.1 summarizes the individual components needed and the quantities actually added.

Table 2.1. Components in Evaporated Cs Eluant Simulant

Component	Species	Source	Targeted Amount (g)	Added Amount (g)
$Al(NO_3)_3$-$9H_2O$	Al	Baker Analyzed	3.19	3.20
H_3BO_3	B	Spectrum Chemical	1.69	1.69
$Ca(NO_3)_2$-$4H_2O$	Ca	Aldrich ACS Reagent	0.454	0.457
Na_2CrO_4-$2H_2O$	Cr	Baker	0.368	0.370
$Cs(NO_3)$	Cs	Research Chemicals	0.499	0.500
$Cu(NO_3)_2$-$2.5H_2O$	Cu	Aldrich ACS Reagent	0.488	0.485
$Fe(NO_3)_3$-$9H_2O$	Fe	Fisher	3.47	3.47
HNO_3 (conc.)	H	Fisher Certified ACS Plus	17*	17*
KNO_3	K	Mallinckrodt	0.621	0.621
$Mn(NO_3)_2$ (50% aqueous solution)	Mn	Baker Analyzed	2.16**	2.16**
$NaNO_3$	Na	Aldrich ACS Reagent	26.2	26.2
$Ni(NO_3)_2$-$6H_2O$	Ni	Aldrich	13.3	13.3
$Pb(NO_3)_2$	Pb	Baker Analyzed	0.105	0.110
Na_2SiO_3-$9H_2O$	Si	Baker	0.904	0.906
$UO_2(NO_3)_2$-$6H_2O$	U	Alfa Ventron	3.25	3.30
HCl (conc.)	Cl	Fisher Certified ACS Plus	1.23*	1.23*

* mL of concentrated acid
** mLs of 50% aqueous solution

With the exception of uranyl nitrate, the individual amounts of all the listed solid sources were placed in a 250 mL glass beaker (wt. = 118.57 grams) together with a magnetic stirring bar (wt. = 4.500 grams). The appropriate amount of the 50% $Mn(NO_3)_2$ solution was added by pipette along with approximately 150 mL of deionized water. Most of the solids dissolved. The concentrated acids were then added to the stirring suspension by pipette yielding a clear solution. The solution was stirred overnight at room temperature and transferred to a radiological fume hood where the uranyl nitrate solid was added. The uranyl nitrate quickly dissolved. The solution was heated and concentrated until just over 100 mL total volume. The beaker was weighed at this point and a small amount of water added until the weight of the simulant approached the targeted weight of 142 g (measured weight = 118.57 + 4.50 + 142 = 265.07 grams). The solution was stirred overnight and allowed to cool to room temperature. The final dark green solution was reweighed the following morning and gave a total solution weight of 264.2 grams.

One mL aliquots of the simulant were weighted to determine the simulant's density. The results are shown in Table 2.2. The DI water control indicates that the pipette was delivering as much as 1% high so a 1% uncertainty probably should be associated with the density of 1.372 g/mL for the simulant. Finally, the simulant (stir bar and all) was transferred to a capped 250 mL polypropylene bottle for storage.

Table 2.2. Density Measurements for Simulant Solution

Aliquot number	Weight of 1 mL aliquot of DI water	Weight of 1 mL aliquot of simulant
1	1.010	1.372
2	1.010	1.373
3	1.005	1.372
4	1.004	-
Ave.	1.007 ± 0.003	1.372

2.1.2 Sr/TRU Precipitate Simulant

The simulated Sr/TRU precipitate was prepared using an AN-107 filtrate simulant created during the Savannah River Technical Center (SRTC) pilot scale testing. The Sr/TRU precipitate was prepared under Test Instruction 29953-082 contained in Appendix C. Table 2.3 lists compositional data from Test Instruction 29953-082 for the AN-107 filtrate simulant obtained from SRTC.

During pilot scale testing at SRTC, the waste was treated with $Sr(NO_3)_2$ and $NaMnO_4$. The resulting precipitate was filtered to remove the solids. A volume of 7.7 L of this AN-107 filtrate simulant was transferred to a reaction vessel at Battelle. To return the material to the original composition, $Fe(NO_3)_3$, $Ca(NO_3)_2$, $Cr(NO_3)_3$, $MnCl_2$, and $Pb(NO_3)_2$ were added. While stirring, 600 mL of 1.0 M $Sr(NO_3)_2$ were added at a rate of 60 mL per minute. Five minutes after the $Sr(NO_3)_2$ addition, 400 mL of 1 M $NaMnO_4$ solution was added at rate of 40 mL per minute. The new solution was stirred for 30 minutes then heated to 50 ±5°C over a period of 1 hour. The solution was then stirred at 50°C for 4 hours. The solution was cooled to 25°C and then filtered. The solids were then washed seven times with ~500 ml of 0.01 M NaOH.

The resulting filtered solids were used as the Sr/TRU Precipitate simulant for rheology testing. Approximately 1200 g of filtered solids were collected. Subsamples (SM-05 and SM-06) were dried at 105°C to determine wt% water. The subsamples SM-05 and SM-06 were found to be 63.4 and 64.0 wt% water respectively. These dried subsamples were then submitted for ICP analysis using an acid digestion to dissolve the solids. Table 2.4 lists the compositional results from the ICP analysis.

Table 2.3 Compositional Data on AN-107 Filtrate Simulant

Analyte	Test 1	Test 2	Units
pH	14	14	N/A
Density	1.272	1.270	kg/L
Al	220	219	mg/L
B	20.8	21.1	mg/L
Ba	<0.02	<0.02	mg/L
Ca	124	128	mg/L
Cd	<0.02	<0.02	mg/L
Cl	1188	1229	mg/L
Co	<0.05	<0.05	mg/L
Cr	0.49	0.53	mg/L
Cu	9.2	12.1	mg/L
F	347	360	mg/L
Fe	35	41	mg/L
K	1553	1523	mg/L
La	1.3	1.2	mg/L
Li	<0.7	<0.7	mg/L
Mg	0.41	0.41	mg/L
Mn	9.4	14.5	mg/L
Mo	20.3	20.5	mg/L
Na	127500	126500	mg/L
Ni	283	285	mg/L
P	318	319	mg/L
Pb	<1.0	<1.0	mg/L
Si	10.2	11.0	mg/L
Sn	<0.4	<0.4	mg/L
Sr	87	95	mg/L
Ti	<0.05	<0.05	mg/L
V	<0.12	<0.12	mg/L
Zn	17	17	mg/L
Zr	0.85	0.79	mg/L
TIC	15320	15699.74	mg/L
TOC	12234	12542.52	mg/L
Formate	5591	5820	mg/L
Nitrate	130202	131115	mg/L
Nitrite	34089	37431	mg/L
Oxalate	2024	1449	mg/L
Phosphate	1534	1549	mg/L
Sulfate	4830	4883	mg/L

Table 2.4. Sr/TRU Precipitate Simulant ICP Results for Dried Solids from Subsamples SM-05 and SM-06. Results are in µg/g of Dried Solids

Analyte	SM-05	SM-06	SM-06 Duplicate	Average
Ca	3.3E+03	3.4E+03	3.4E+03	3.4E+03
Cr	5.8E+02	5.9E+02	6.0E+02	5.9E+02
Fe	2.6E+04	2.7E+04	2.7E+04	2.7E+04
Mn	1.6E+05	1.6E+05	1.6E+05	1.6E+05
Na	3.6E+04	4.0E+04	4.0E+04	3.9E+04
P	7.3E+02	6.5E+02	7.5E+02	7.1E+02
Pb	1.2E+03	1.1E+03	1.2E+03	1.2E+03
Sr	3.9E+05	4.0E+05	3.9E+05	3.9E+05

2.2 Evaporation and Settling Study

The actual waste samples used in this testing were prepared under conditions similar to those anticipated in the River Protection Project Waste Treatment Plant flow sheet. Both the AZ-102 and C-104 wastes were received for this task following ultrafiltration, caustic leaching and solids washing steps.

The AZ-102 feed was received for this task containing 9.54 wt% solids. At this solids concentration, no standing liquid was observed in the AZ-102 feed. The C-104 feed was received with 20 wt% solids and contained roughly 15 vol% supernatant liquid. Following receipt, each waste was partitioned into four subsamples. Three of the subsamples were adjusted to the target solids concentrations (5 wt%, 15 wt%, and 25 wt%) and the fourth was retained for the mixing/aging study. The AZ-102 5 wt%, C-104 5 wt% and 15 wt% solids samples were prepared by diluting with deionized water. The remaining samples were initially concentrated by decanting standing liquid. Additional water was later evaporated by flowing air over the sample surface at ambient temperature.

Following the evaporation step, the samples were hand stirred with a spatula and subsamples were transferred in duplicate to 10 ml graduated centrifuge cones. Additional subsamples were removed for rheological testing. The rheological testing is described later in this report. The cones were agitated and allowed to settle for 3 days at ambient hot cell temperature (nominally 34°C). During this settling period, the volume of settled solids was recorded as a function of time. The total mass and volume was also recorded and used to calculate the density of the bulk slurries at 34°C. The samples were then transferred to an oven at 50°C for ~12 hours. The cones were then agitated and returned to the oven and allowed to settle for an additional 3 days. During this settling period, the volume of settled solids was recorded as a function of time. The total mass and volume was also recorded and used to calculate the density of the bulk slurries at 50°C.

The material in the centrifuge cones was then returned to the respective bottles along with material used in rheological testing. In most cases deionized water was used to aid the transfer. The samples were then readjusted to the respective solids contents (5, 15 and 25 wt%) by decanting and evaporation using a flow of ambient air. Simulated secondary waste products were added to the samples as detailed in Appendix A to Test Plan 29953-073 contained in Appendix B. The masses of the samples of secondary waste products are listed in Table 2.5. The samples were then stirred using an overhead mixer for approximately 30 minutes. A four bladed impeller, 3.8 cm in diameter, was used at a rotational rate of ~8000 RPM.

Following secondary waste product addition to the C-104 samples, subsamples were transferred in duplicate to 10 ml graduated centrifuge cones. Additional subsamples were removed for rheological testing. The cones were agitated and allowed to settle for 3 days at ambient hot cell temperature (nominally 34°C). During this settling period, the volume of settled solids was recorded as a function of time. The samples were then transferred to an oven at 50°C for ~12 hours. The cones were then agitated and returned to the oven and allowed to settle for an additional 3 days. During this settling period, the volume of settled solids was recorded as a function of time. The samples were then cooled to ambient temperature and centrifuged for approximately 10 minutes at ~1000 times the force of gravity (1000 x g). The total mass and volume were then recorded and used to calculate the density of the bulk slurries at 34°C. The samples where then placed in an oven at 50°C for approximately 12 hours and the density measurements were repeated.

The material in the centrifuge cones (AZ-102 evaporated feeds, and C-104 evaporated feeds with secondary waste products) were then returned to the respective sample bottles using water to aid in the transfer. Material from rheological testing was also returned to the respective bottles. Given the presence of dissolved solids in the secondary wastes, excess supernatant was not decanted from the C-104 feeds. The C-104 samples were readjusted to their appropriate water contents though evaporation by passing a flow of air over their surfaces. The AZ-102 feeds were readjusted to their appropriate water contents though decanting and evaporation. Glass formers were added to the samples as detailed in Appendix A to Test Plan 29953-073 contained in Appendix B. The mass of the evaporated slurries (before addition of secondary waste products) and mass of glass formers are listed in Table 2.6 and Table 2.7 for AZ-102 and C-104 samples respectively. The samples were then stirred using an overhead mixer for approximately 15 minutes. A four bladed impeller, 3.8 cm in diameter, was used at a rotational rate of ~8000 RPM.

Given the high viscosity and yield point for the AZ-102 25 wt% sample, glass formers were not added to this sample. Instead, the AZ-102 25 wt% sample was diluted to 20wt% solids, and the rheology of this new concentration was examined at 25°C.

Following glass former addition, subsamples of both C-104 (5, 15 and 25 wt%) and AZ-102 feeds (5 and 15 wt% only) were transferred in duplicate to 10 ml graduated centrifuge cones. Additional subsamples were removed for rheological testing. The cones were agitated and allowed to settle for 3 days at ambient hot cell temperature (nominally 34°C). During this settling period, the volume of settled solids was recorded as a function of time. The samples were then transferred to an oven at 50°C for ~12 hours. The cones were then agitated and returned to the oven and allowed to settle for an additional 3 days. During this settling period, the volume of settled solids was recorded as a function of time. The samples were then cooled to ambient temperature and centrifuged for approximately 10 minutes at ~1000 times the force of gravity (1000 x g). The total mass and volume were then recorded and used to calculate the density of the bulk slurries at 34°C. The samples where then placed in an oven at 50°C for approximately 12 hours and the density measurements were repeated.

Table 2.5. Mass of Samples and Secondary Waste Products For C-104 Tests

Secondary Waste	5 wt%	15 wt%	25 wt%
Slurry Mass (g)	102.7	68.1	68.0
Simulated Sr/TRU (g)	6.1	12.3	20.3
Simulated Cs Eluant (g)	0.74	1.44	2.45

Table 2.6. Mass of Samples, Glass Formers and Secondary Waste in Grams Added to AZ-102 Samples. Sample 15 wt%-2 Was Used for Mixing and Aging Study

Sample	5 wt%	15 wt%	15 wt%-2
Slurry Mass	79.83	77.59	86.70
Sr/TRU Precipitate (25 wt% solids basis)	1.841	5.367	5.997
$Na_2B_4O_7$+$10H_2O$	1.031	3.01	3.359
LiOH+H_2O	1.486	4.33	4.842
Na_2SiO_3+$5H_2O$	3.075	8.96	10.017
SiO_2 (Sil-co-Sil 75)	3.807	11.1	12.402
Sugar (Granular)	0.128	0.374	0.418

Glass formulations in this report for AW-101 and AN-107 are considered to be proprietary by VSL/Duratek

Table 2.7. Mass of Samples and Glass Formers Added to C-104 Samples

Sample	5 wt%	15 wt%	25 wt%-1	25 wt%-2
Slurry Mass (g)	102.7	68.1	68.0	68.2
Sr/TRU Precipitate Simulant (26.8 Wt% Solids Basis)	6.1	12.3	20.3	20.5
Cs Eluant Simulant	0.737	1.436	2.451	NA
$Na_2B_4O_7$+$10H_2O$	3.723	7.406	12.325	12.361
LiOH+H_2O	2.362	4.699	7.820	7.843
Na_2CO_3	0.226	0.450	0.749	0.751
SiO_2 (Sil-co-Sil 75)	7.463	14.848	24.710	24.782
ZnO (KADOX-920)	0.316	0.630	1.048	1.051

NA, Cs Eluant not added to C-104-25-2

2.3 Shear Stress Versus Shear Rate Analysis (Rheology)

As described in Section 2.2, shear stress versus shear rate analyses were conducted on the evaporated feeds as well as following addition of secondary waste products and glass formers. These measurements were conducted at 25°C and 50°C using a Haake M5. The M5 head was modified for operation in a high radiation field by relocating the printed circuit boards from the head to a remote box as well as extending the signal cables and adding a secondary heat exchanger to the recirculating loop. Other minor modifications were made to the water jacket and measuring sensors to aid in operations using master-slave manipulators.

Measurements were made using an SV I sensor. The recommended range for this sensor is from 300 to 100,000 cP with a maximum torque of 1240 Pa and a maximum shear rate of 445 s^{-1}. This was found to be an appropriate sensor for the 15 and 25 wt% samples, but was not sensitive enough for some of the 5 wt% samples. However, the SV I was used for all samples since there was insufficient material available to use the more sensitive MV sensors. The SV I requires approximately 10 ml while the MV I requires 40 ml.

During this work a 95.5 cP Brookfield viscosity standard was used to check the calibration of the instrument. Calibration checks were performed a minimum of once a month when the instrument was in use. All calibration checks were within 10% of the certified value.

Shear stress versus shear rate rheograms were obtained by measuring the shear stress produced at a specific shear rate. The increasing shear rate curve was generated by gradually increasing the shear rate from 0 to 300 s^{-1} over a 2 minute period. The decreasing shear rate curve was then generated by reducing the shear rate back down to 0 s^{-1} over 2 minutes. The shear rate analysis was conducted again at least once with the same sample still in the instrument. A difference between duplicate runs would indicate potentially unusual behavior in the samples including (but not limited to) settling of the solids within the instrument, the sample being effected by shearing in the instrument, or water loss through evaporation.

2.4 Mixing and Aging Study

As described in Section 2.2, the initial AZ-102 and C-104 material received for this work were each partitioned into 4 subsamples. Three of the subsamples were used for evaporation and settling studies while one sample from each tank was retained for the mixing and aging studies. The retained AZ-102 sample was adjusted to 20 wt% solids and subjected to a shear stress versus shear rate test prior to the mixing and aging study. The C-104 sample was not subjected to any testing prior to the mixing and aging study.

The AZ-102 sample was diluted from 20wt% solids to the 15 wt% using deionized water. The C-104 sample was evaporated to 25 wt% solids. Both samples were then placed in 250 ml round bottom flasks with side tubes.

Both samples were stirred using an overhead mixer while glass formers were added to the AZ-102 sample, and glass formers and secondary waste products were added to the C-104 sample. The flasks were then sealed using a Teflon stirrer bearing. The sample was then continually stirred for 1 week using a 1-inch (2.54 cm) diameter blade at approximately 400 rpm. The rate of the mixer was checked approximately every day and adjusted as needed to maintain the rate of 400 rpm. The volume of the slurries was approximately 6.1 in^3 (100 ml). The 400 rpm mixing rate provides roughly the same energy per volume as anticipated in the River Protection Project Waste Treatment Plant. The following equation (provided by BNFL) was used to calculate the proper rotational rate:

$$N^3 = 1.85 \times 10^7 \, V/D_i^5$$

For this equation, N is the impeller rotational rate in rpm, V is the sample volume in cubic inches and D_i is the impeller diameter in inches.

Samples of each of the slurries were removed from the mixing vessel after 1 hour, 1 day and 1 week. These samples were immediately analyzed for shear stress versus shear rate at 25°C. These analyses were conducted as described in section 2.3.

After one week of mixing, the AZ-102 and C-104 slurries were transferred to graduated glass jars. The sample was left to settle undisturbed for 1 week. During the settling time, the samples were monitored for any gas retention and releases. Visual observations were supplemented with time lapse video. No gas bubbles were observed in these samples during this study.

After one week of settling, the standing liquid was removed from the slurries, and the settled solids were immediately analyzed for shear stress versus shear rate at 25°C. As described in section 2.3, the analyses were conducted by increasing the shear rate from 0 to 300 s^{-1} over 2 minutes generating the increasing shear rate curve, and then back down to 0 s^{-1} over 2 minutes generating the decreasing curve.

3.0 Experimental Results

This section details the results of tests conducted on actual AZ-102 and C-104 samples following adjusting to target feeds with solids concentrations of 5, 15, and 25 wt%. The initial AZ-102 and C-104 feeds were 9.54 and 20 wt% solids respectively. The feeds were adjusted to the targets using a combination of decanting supernatant liquid, evaporation at ambient temperature (~34°), and addition of deionized water. Following addition of secondary waste products and glass formers the resulting melter feed slurries had a higher solids content. However, in this report, samples will be referred to according to the target solids content for the evaporated feed (e.g. the feed evaporated to 25 wt% solids is still referred to as the 25 wt% solids slurry even after the addition of dry glass formers).

3.1 Density and Settling Study

The densities of the evaporated samples, samples with secondary waste products and, samples following the addition of glass formers are provided in Table 3.1.

As expected, the densities of the initial feeds prior to glass former or secondary waste product additions increase with increasing sample concentration and decreases very slightly with temperature. No temperature trend is seen in the C-104 samples following secondary waste product additions. No significant temperature trends are seen in either the AZ-102 or C-104 samples following glass former additions. For untreated Hanford tank waste slurries, temperature related density trends are usually observed and are the result of solids dissolution. The lack of temperature trends suggests that there is no significant solids dissolution occurring between 34 and 50°C. This is not surprising since the AZ-102 and C-104 solids were washed and leached prior to this feed testing.

As described in Section 2.2, the graduated centrifuge cones were agitated and then allowed to settle at ambient (approximately 34°C) and 50°C over a 2 to 3 day period. Due to the Hanford brush fire during the week of June 25, 2000, the settling study on the C-104 samples with glass formers was terminated after 30 hours.

For most samples, the solids settled leaving a clear supernatant above with a distinct interface between the settling solids and the clarified supernatant. The level of this interface in the graduated centrifuge cone was measured as the volume of settled solids and divided by the total sample volume to calculate the volume percent (vol%) settled solids. Table 3.2 lists the vol% settled solids for the samples in this study. The 15 and 25 wt% AZ-102 samples were not examined since these samples did not settle to form a standing liquid layer after one week undisturbed in the primary sample jars. The standing liquid in the 5 wt% C-104 evaporated feed did not clarify over the three days of settling and an accurate measure at 34°C could not be made. At 50°C, a settled solids layer at ~8 vol% was observed for these duplicate samples.

Table 3.1. Density of AN-107 and AW-101 Samples at 25°C and 50°C With and Without Addition of Glass Formers in g/ml.

		Evaporated Feed		With Secondary Waste [b]		With Glass Formers [a]	
Feed	Sample	34°C	50°C	34°C	50°C	34°C	50°C
5 wt% AZ-102	Primary	1.03	1.02	NM	NM	1.08	1.06
	Duplicate	1.05	1.04	NM	NM	1.09	1.08
	Average	1.04	1.03	NM	NM	1.09	1.07
	Relative % Difference	2%	2%	NM	NM	1%	2%
15 wt% AZ-102	Primary	1.15	1.11	NM	NM	1.23	1.23
	Duplicate	1.13	1.12	NM	NM	1.22	1.22
	Average	1.14	1.12	NM	NM	1.23	1.23
	Relative % Difference	2%	1%	NM	NM	1%	1%
25 wt% AZ-102	Primary	1.26	1.20	NM	NM	NM	NM
	Duplicate	1.22	1.23	NM	NM	NM	NM
	Average	1.24	1.22	NM	NM	NM	NM
	Relative % Difference	3%	2%	NM	NM	NM	NM
5 wt% C-104	Primary	0.99	0.99	1.01	1.01	1.09	1.08
	Duplicate	0.99	0.99	1.01	1.01	1.07	1.07
	Average	0.99	0.99	1.01	1.01	1.08	1.07
	Relative % Difference	1%	1%	0%	0%	2%	0
15 wt% C-104	Primary	1.04	1.02	1.12	1.12	1.30	1.27
	Duplicate	1.06	1.06	1.10	1.10	1.33	1.27
	Average	1.05	1.04	1.11	1.11	1.32	1.27
	Relative % Difference	2%	4%	2%	2%	2%	0
25 wt% C-104	Primary	1.10	1.08	1.20	1.20	1.41	1.40
	Duplicate	1.14	1.12	1.17	1.17	1.41	1.40
	Average	1.12	1.10	1.19	1.19	1.41	1.40
	Relative % Difference	4%	4%	3%	3%	0%	0

a. C-104 density measurements made after addition of secondary waste products and glass formers
b. secondary waste products were not added to AZ-102 slurries

Table 3.2. Volume Percent Settles Solids for the AZ-102 Samples

		Evaporated Feed		With Secondary Waste		With Glass Formers[a]	
Feed	Sample	34°C	50°C	34°C	50°C	34°C	50°C
5 wt% AZ-102	Primary	58	60	NM	NM	49	59
	Duplicate	56	62	NM	NM	51	58
	Average	57	61	NM	NM	50	58.5
	Relative % Difference	4%	3%	NM	NM	4%	2%
5 wt% C-104	Primary	50[b]	7.6	19	24	26	32[c]
	Duplicate	48[b]	8.3	19	22	24	31[c]
	Average	49[b]	7.95	19	23	25	31.5[c]
	Relative % Difference	4%	9%	0%	9%	8%	3%
15 wt% C-104	Primary	67	73	76	82	80	89[c]
	Duplicate	69	78	71	77	79	88[c]
	Average	68	75.5	73.5	79.5	79.5	88.5[c]
	Relative % Difference	3%	7%	7%	6%	1%	1%
25 wt% C-104	Primary	92	94	NM	NM	NM	NM
	Duplicate	93	99	NM	NM	NM	NM
	Average	92.5	96.5	NM	NM	NM	NM
	Relative % Difference	1%	5%	NM	NM	NM	NM

a. C-104 Glass formers were added to C-104 samples after addition of secondary waste products
b. Incorrect interface measured due to cloudy supernatant layer. Supernatant in 5 wt% C-104 initial feed samples did not clear after 3 days of settling
c. Only allowed to settle for 30 hours. Slope of settling curves (Figure 3.13) strongly suggest final vol% settled solids at 50°C would have been higher than at 34°C.

Surprisingly for every sample examined, the samples at 50°C did not compact as well as the same samples at 34°C. The trend of increasing settled solids volume with temperature was between 4 and 8 vol%. This is probably the result of the test methodology. The cones were loaded and agitated prior to the 34°C study. The samples were then allowed to settle, and then the cones were agitated again before the 50°C study. This agitation probably broke some of the aggregate structure in the settled solids resulting in a slightly looser settled solids volume. This is supported by shear stress versus shear rate tests in which the viscosity of the slurries decrease over duplicate analyses with the same sample. Future settling studies should include a repeat of the 34°C settling study after the 50°C test to confirm this behavior.

For the AW-101 and AN-107 low activity waste (LAW) samples reported in a previous study (Bredt and Swoboda, 2000) the 50°C tests contained a smaller vol% settled solids compared to the duplicates at 25°C. This trend of decreasing vol% settled solids with increasing temperature for the LAW samples was attributed to either the dissolution of soluble species leading to a lower volume of solids or a decrease in the viscosity and yield stress resulting in greater compaction of the solids. These factors should not affect the C-104 and AZ-102 wastes since these feeds contain little to no soluble solids and no significant change in viscosity or yield stress was observed between 34 and 50°C.

As expected, the data shows the vol% settled solids increased with solids content. The vol% settled solids increased with increasing solids contents in the evaporated feeds (5 wt% < 15 wt% < 25 wt%), and the vol% settled solids increased with the addition of solids from secondary waste products and glass formers. This trend is not surprising since the initial tank solids and Sr/TRU secondary waste products were rinsed during previous testing, removing soluble solids. In addition, all of the glass formers with the exception of sugar and boric acid are highly insoluble.

Figure 3.1 and 3.2 plots the vol% settled solids (interface height/sample height x 100%) versus time for the 5 wt% AZ-102 evaporated feed. Figure 3.1 uses a linear scale while Figure 3.2 is semi-log scale. The linear scale is provided to show stability in the final vol% settled solids while the semi-log scale is useful for examining the behavior over the first few hours. Both duplicates reached a stable settled solids layer at 34 and 50°C within approximately 30 hours. Figure 3.3 plots the settling rate as a function of time using a log scale. The settling rate was calculated by dividing the change in settled solids height by time. Minor parallax errors can be magnified by this calculation and result in significant scatter. To minimize scatter the data plotted in Figure 3.3 have been smoothed using a four point moving average.

Three settling mechanisms are generally observed for this type of sample matrix: free settling, hindered settling, and compression settling. Free settling is settling of discrete particles or flocculated particles without interaction from other particles or the wall. Free settling is denoted by a linear decrease in vol% settled solids over time as the particles fall with fixed velocities. Since the settling rate is simply the slope of the vol% settled solids versus time plot time, a linear decrease in settling velocity would be seen as a flat portion of the settling rate versus time plot. For flocculating systems, the velocity generally increases as the mass of the particles increase, although particle shape changes can also decrease the settling velocity. Hindered settling occurs when the when particle-particle and particle-wall interactions effect the settling velocities. In an ideal system, hindered settling is denoted by the break from a linear decrease in vol% settled solids with time (i.e. a decrease in the settling rate). Compression settling is the final portion of the vol% settling curve as the system approaches the stable value. Two or even all three of these mechanisms usually occur simultaneously.

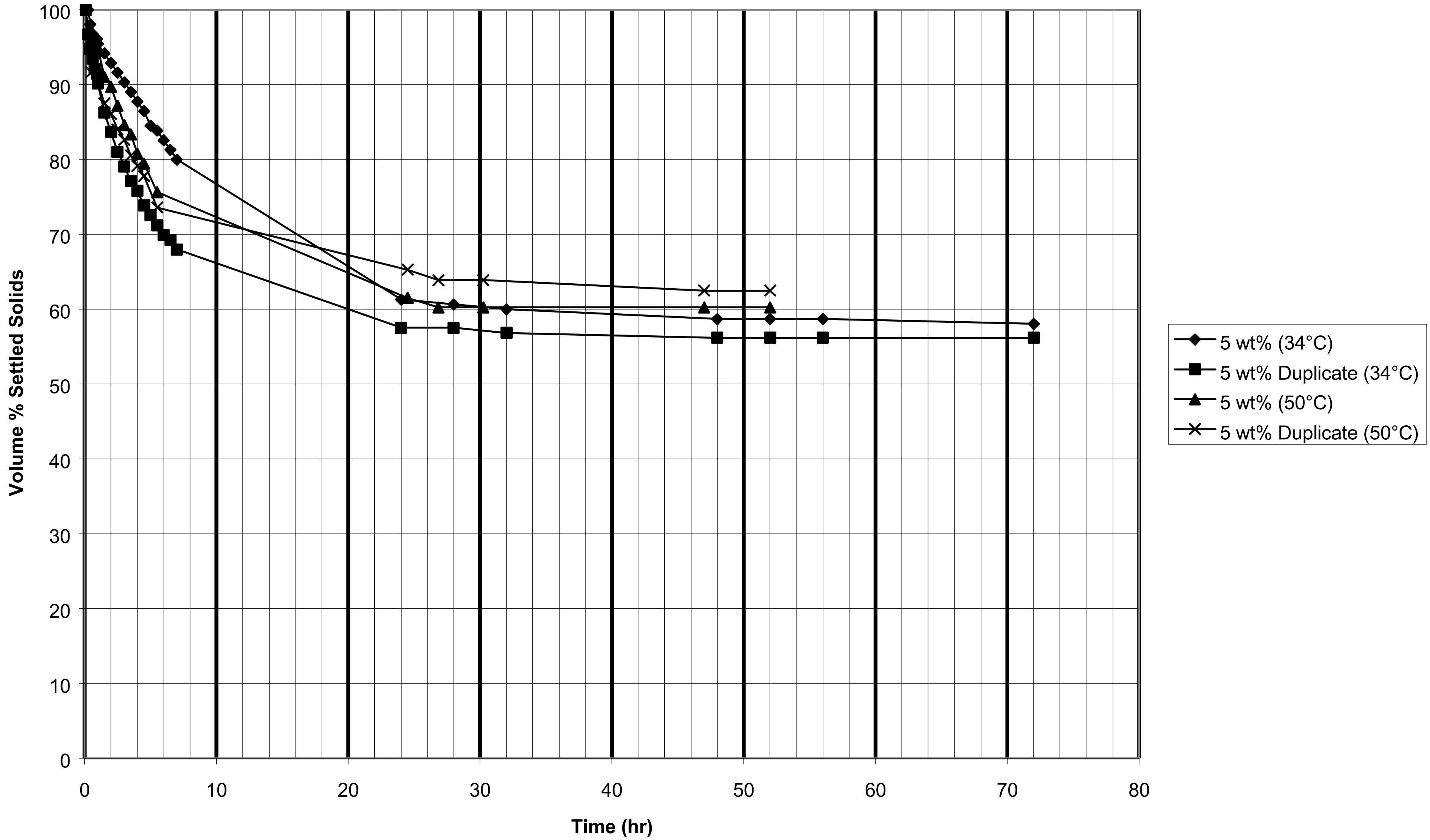


Figure 3.1. Volume Percent Settled Solids Versus Time for AZ-102 Evaporated Feed Using a Linear Scale

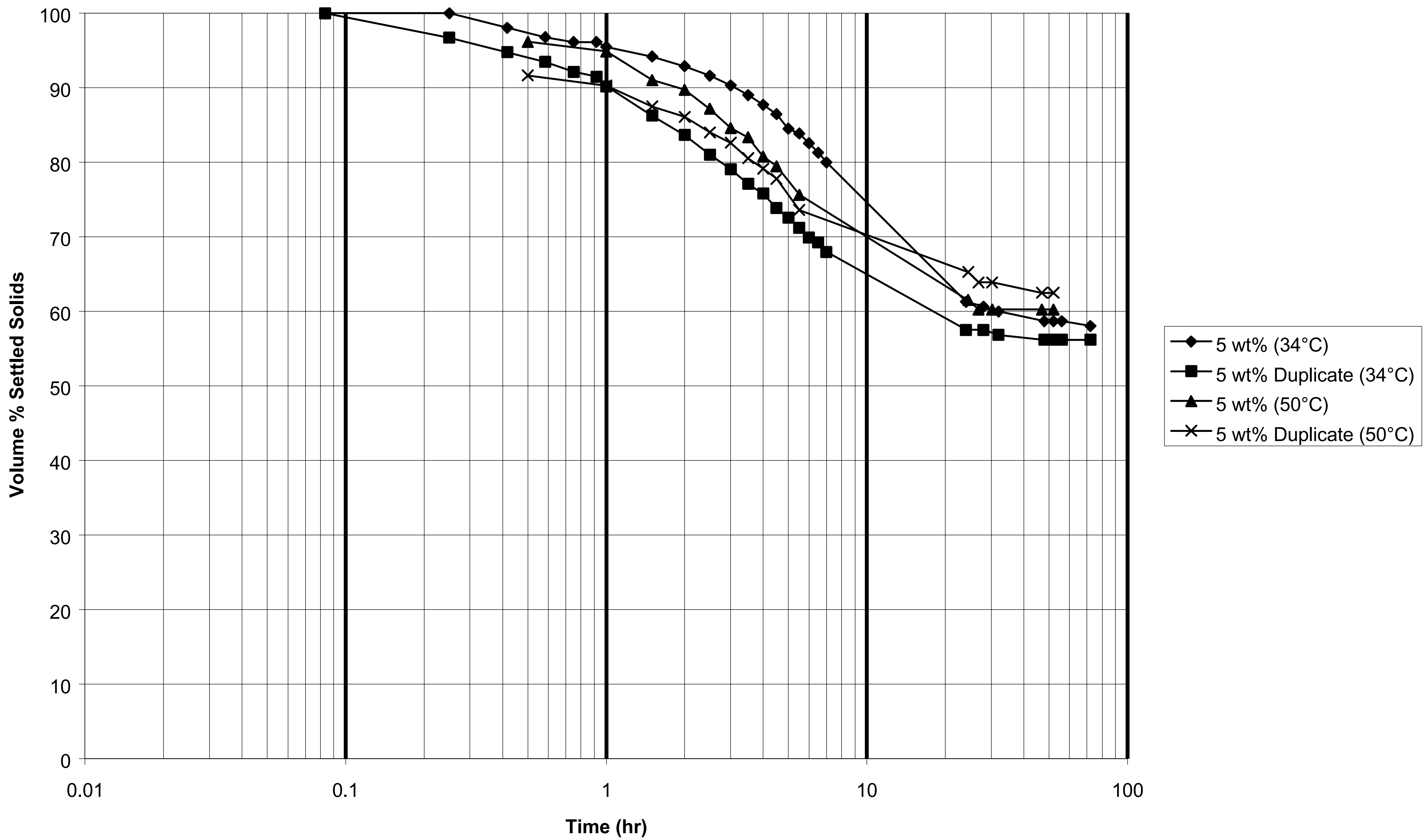


Figure 3.2. Volume Percent Settled Solids Versus Time for AZ-102 Evaporated Feed Using a Semi-Log Scale

The 34°C samples in Figure 3.3 show an initially constant settling rate between 0.001 and 0.004 cm/min for the first 18 minutes. This region was not observed in the samples at 50°C because the 50°C samples were only monitored after 30 minutes of settling. This region with constant settling rate is probably a combination of free as well as some hindered settling. Free settling of the smaller particles is dominating the behavior of the observed solids interface. However, the majority of the solids are at a much higher concentration at the bottom of the column where hindered settling is the dominant mechanism.

After approximately 18 minutes of settling, the settling rate for all of the samples drop steadily as seen in Figure 3.3. This drop indicates the end of any free settling, and a transition to hindered and compression settling. This region of the settling curve is probably a mixture of compression settling and hindered settling. From this settling study it is difficult to determine the transition from the combination of both hindered and compressive settling to only compression settling. This could be possible in future work by use of a microbalance technique in which the solids settle onto a balance pan suspended in the solution.

Figures 3.4 and 3.5 plots the vol% settled solids versus time for the AZ-102 5 wt% samples after glass former addition. Figure 3.6 plots the settling rates versus time. The same initial free settling behavior is seen in the material after glass former addition as before glass former addition. Comparing Figure 3.3 to 3.6 one sees the samples with glass former exhibit constant settling rates for approximately the first 30 minutes, the rate then drops as hindered and compressive settling become the dominant behaviors. The settling rates for the AZ-102 material increases by approximately a factor of four following glass former addition, from between 0.001-0.003 cm/min to 0.005-0.11 cm/min. This shows some flocculation of the AZ-102 sludge particles with the glass formers.

Settling data for the C-104 samples are similarly presented in Figures 3.7 through 3.15. As with the AZ-102 samples, no temperature trends are observed in the C-104 settling rates between 34 and 50°C. Comparing Figures 3.9 and 3.12, one sees addition of secondary waste products greatly increases the initial settling rate. The initial rate for the evaporated feeds (Figure 3.9) are between 0.0004 and 0.004 cm/min. The rates following secondary waste product addition increases by a factor of 10 to between 0.004 and 0.04 cm/minute. This increase is most likely the result of flocculation of the sludge particles with the secondary waste. Most notably, the supernatant in the 5 wt% initial feed remained cloudy throughout the settling period. Following addition of secondary waste, the 5 wt% samples settled with clarified supernatant. Similar high settling rates (between 0.004 and 0.04 cm/minute) were observed for the samples after glass former addition, see Figure 3.15. Since the glass formers were added after secondary waste products, it is not known whether glass formers alone would induce similar flocculation.

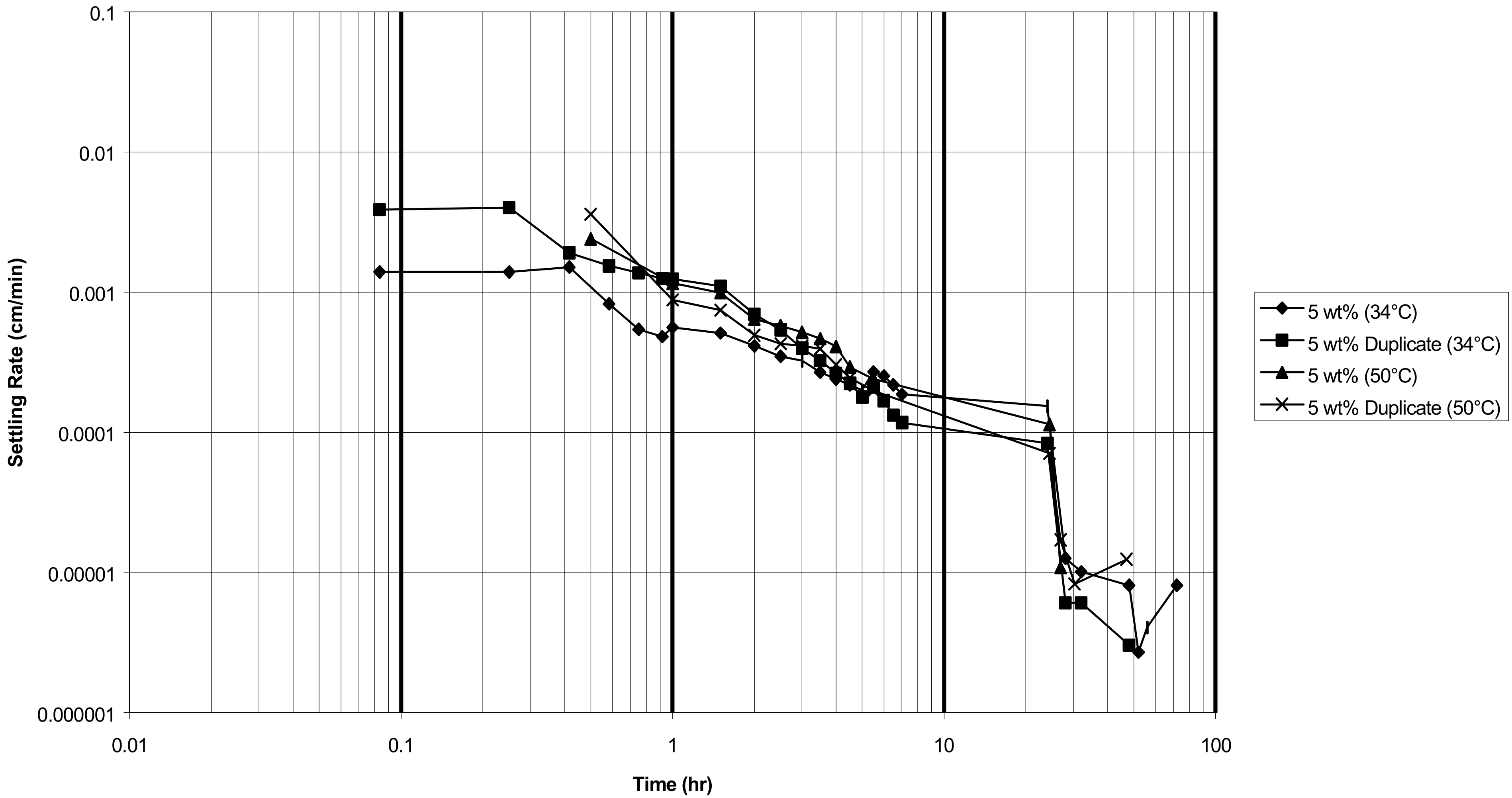


Figure 3.3. Settling Rates for AZ-102 Evaporated Feed

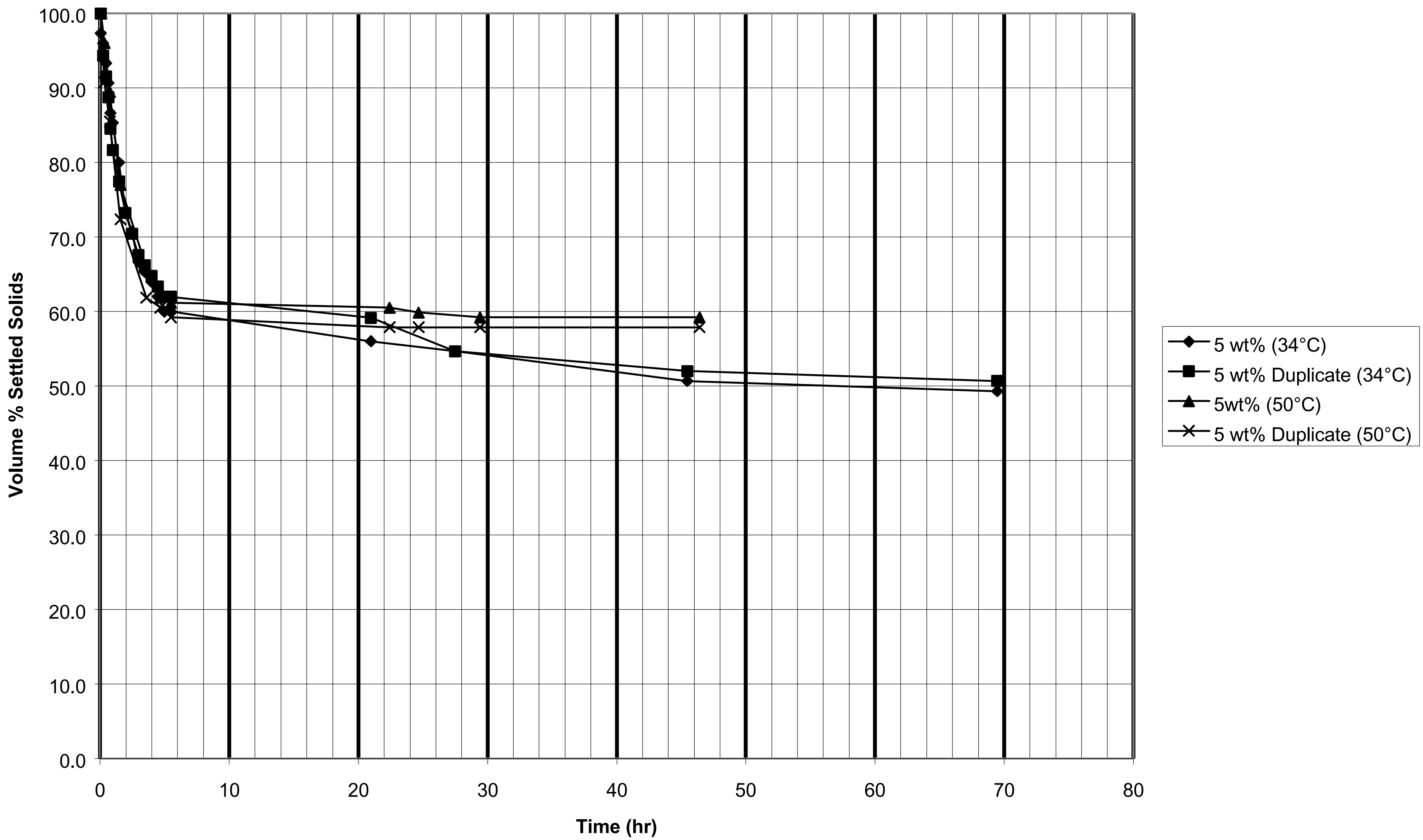


Figure 3.4. Volume Percent Settled Solids Versus Time for AZ-102 Evaporated Feed With Glass Formers Using a Linear Scale

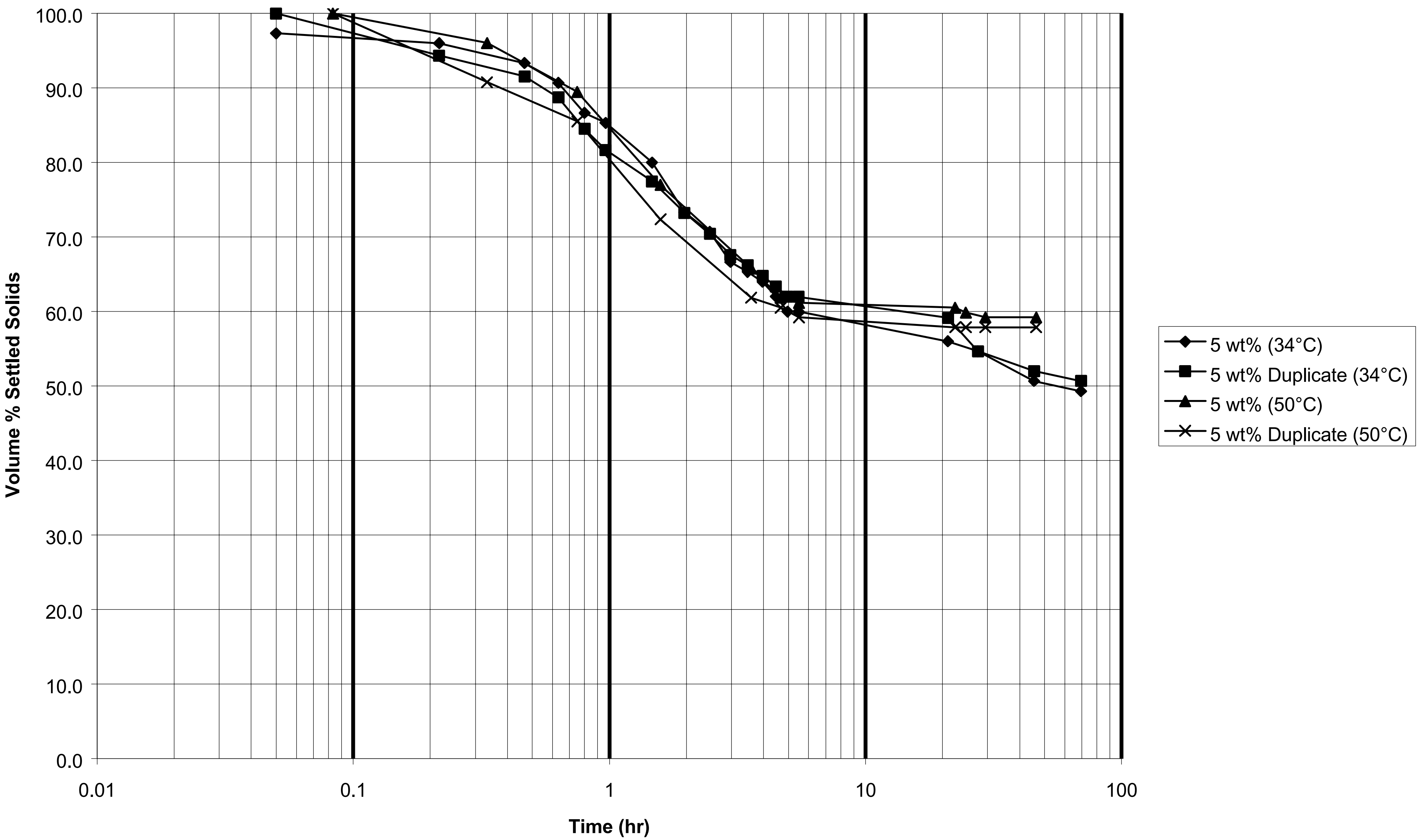


Figure 3.5 Volume Percent Settled Solids Versus Time for AZ-102 Evaporated Feed With Glass Formers Using a Semi-Log Scale

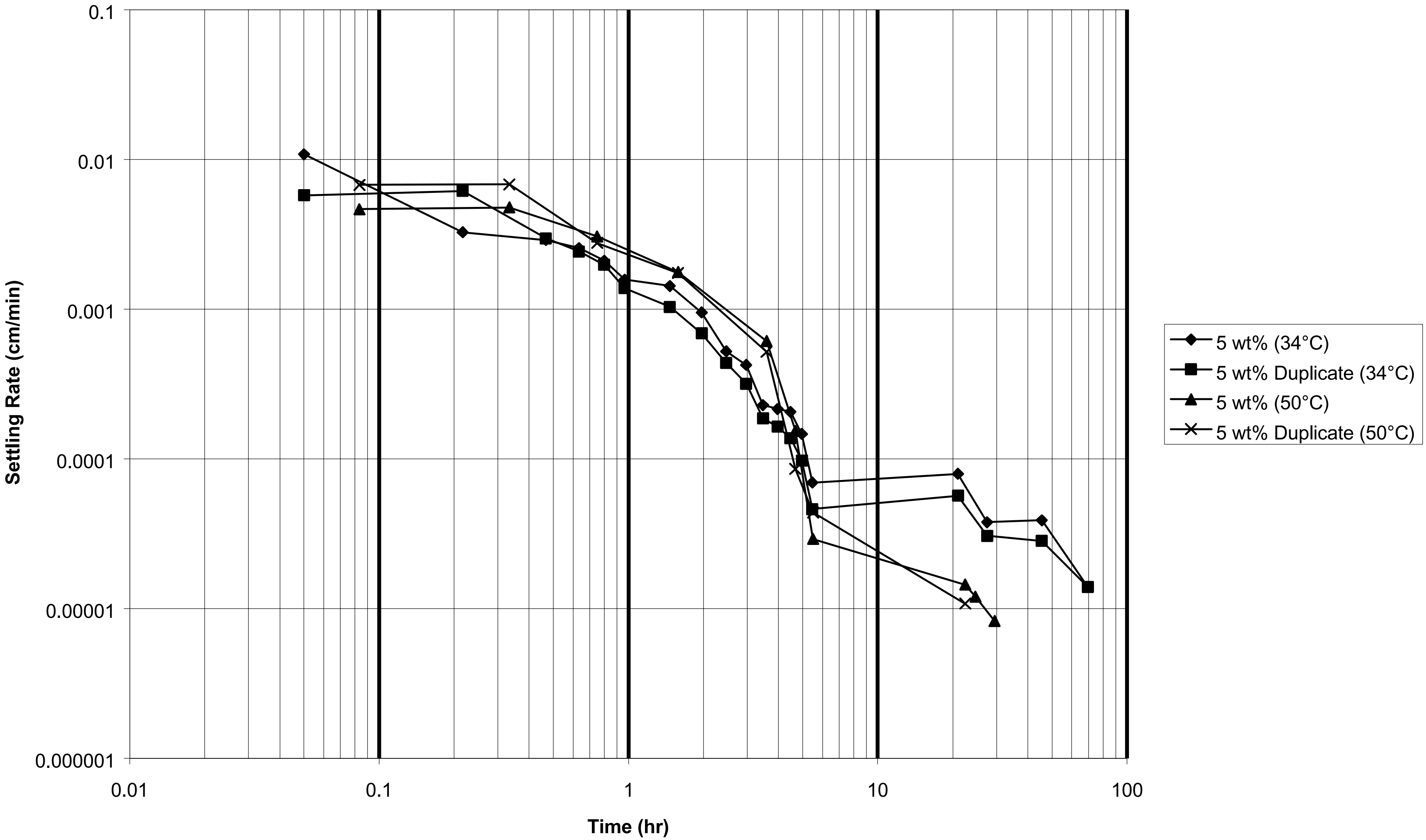


Figure 3.6. Settling Rates for AZ-102 Evaporated Feed with Glass Formers

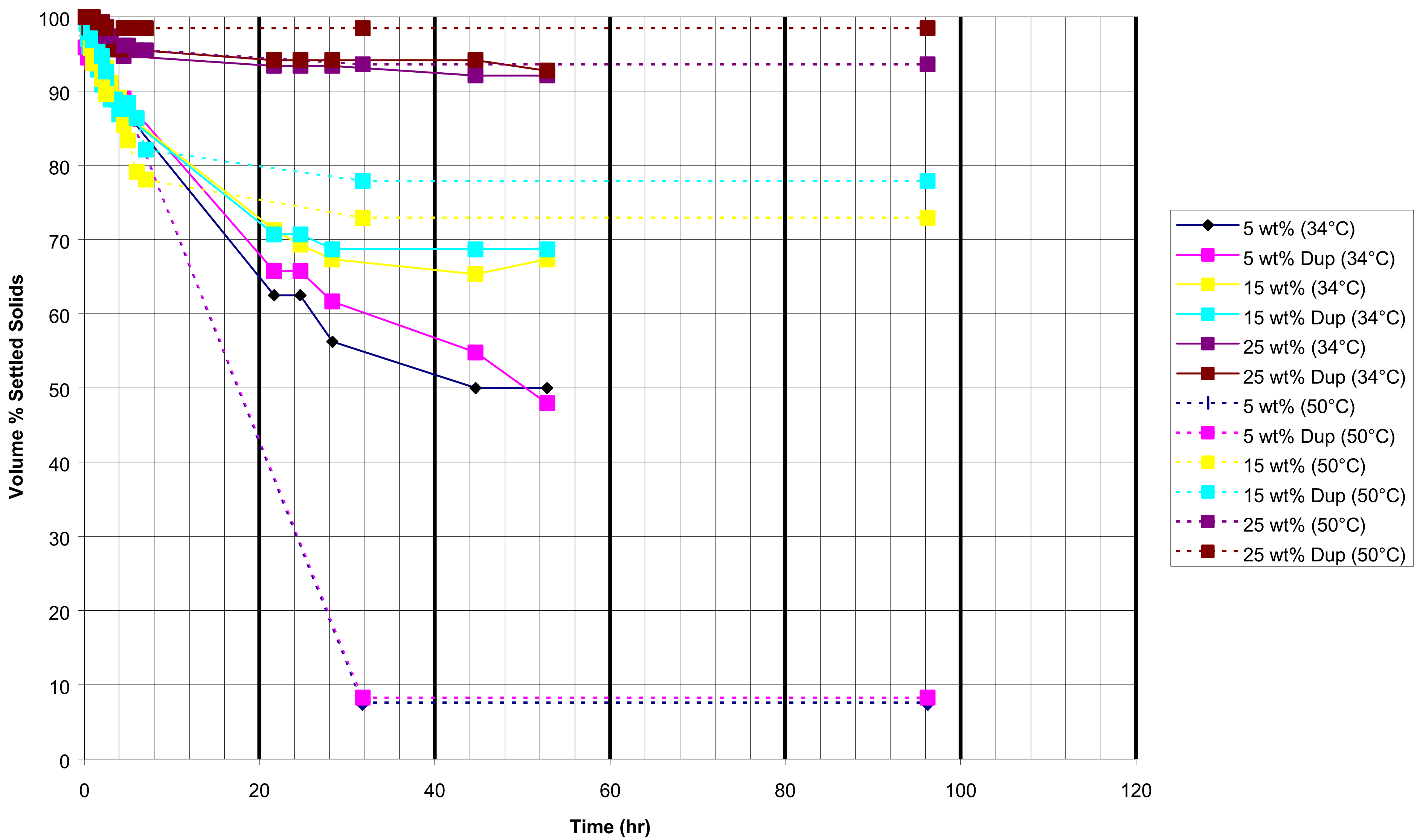


Figure 3.7. Volume Percent Settled Solids Versus Time for C-104 Evaporated Feed Using a Linear Scale

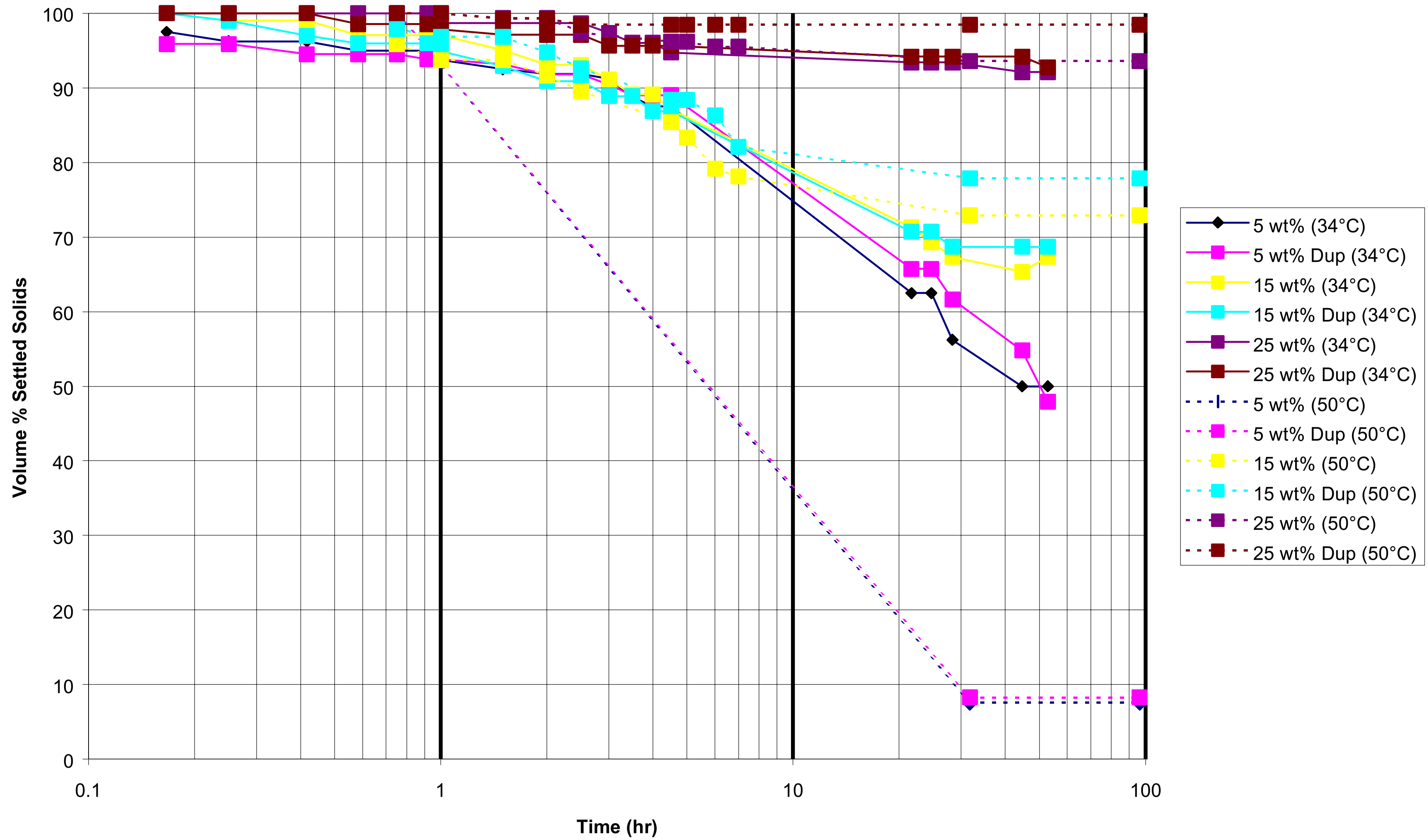


Figure 3.8. Volume Percent Settled Solids Versus Time for C-104 Evaporated Feed Using a Semi-Log Scale

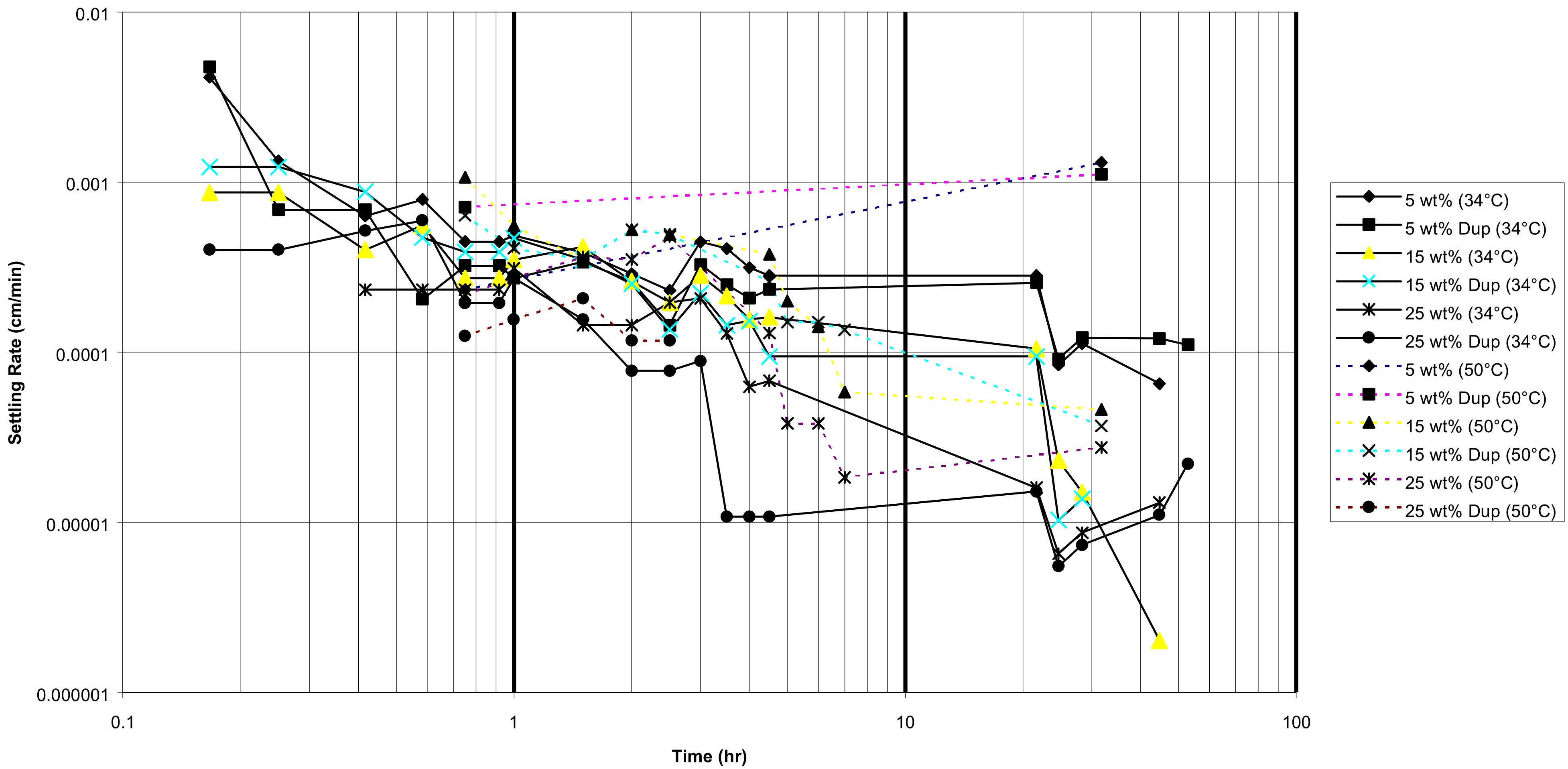


Figure 3.9. Settling Rates for C-104 Evaporated Feed

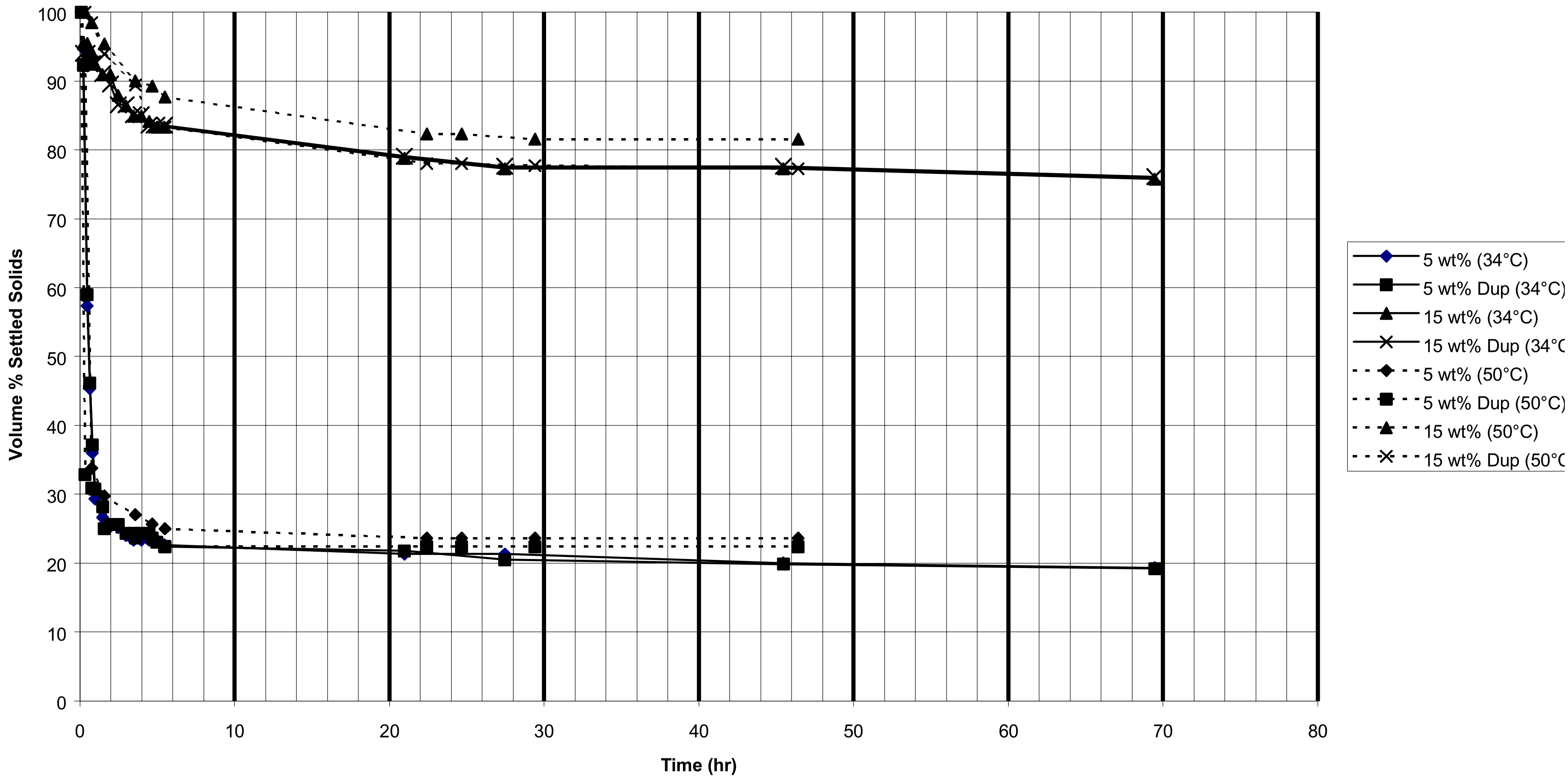


Figure 3.10. Volume Percent Settled Solids Versus Time for C-104 Evaporated Feed With Secondary Waste Using a Linear Scale

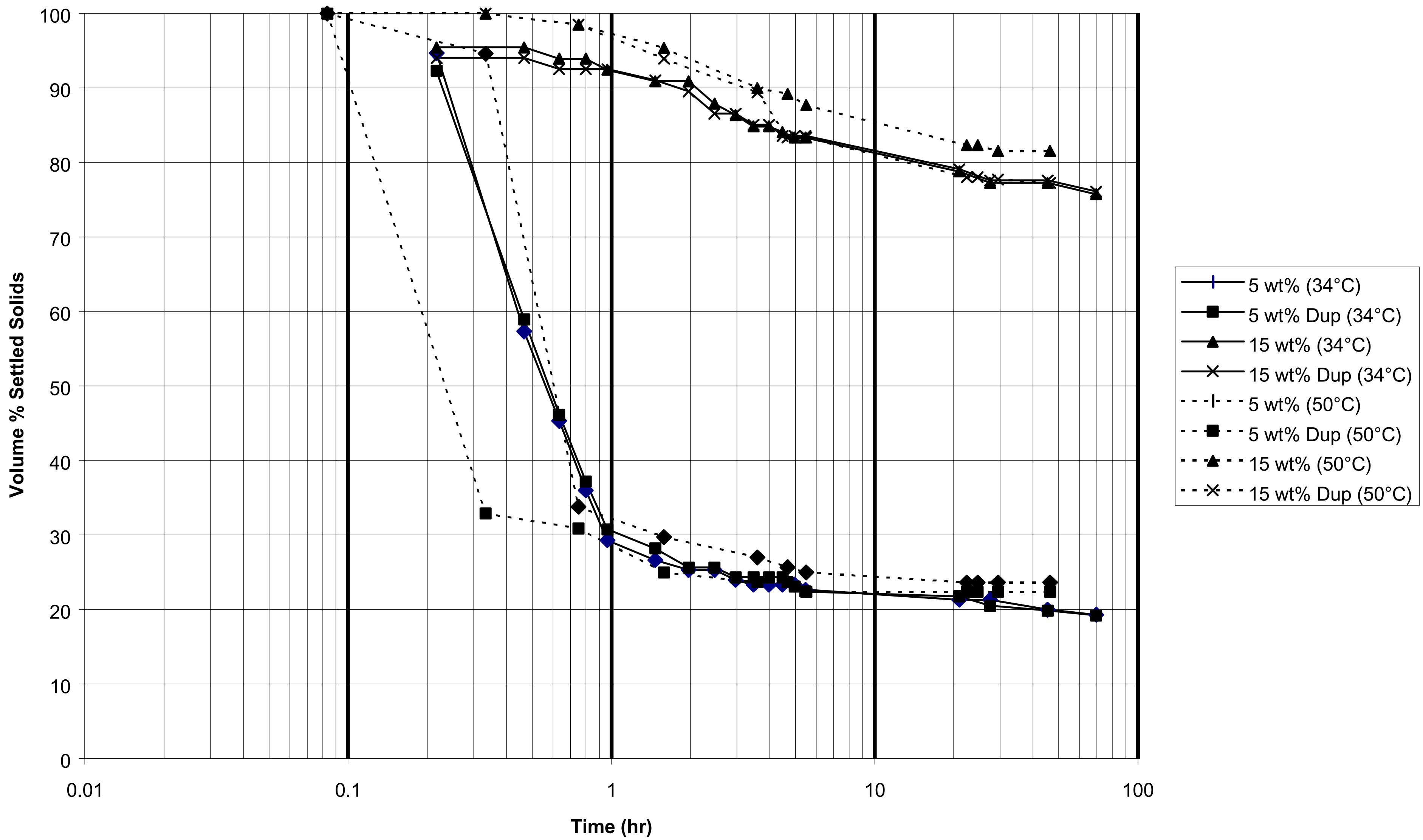


Figure 3.11. Volume Percent Settled Solids Versus Time for C-104 Evaporated Feed With Secondary Waste Using a Semi-Log Scale

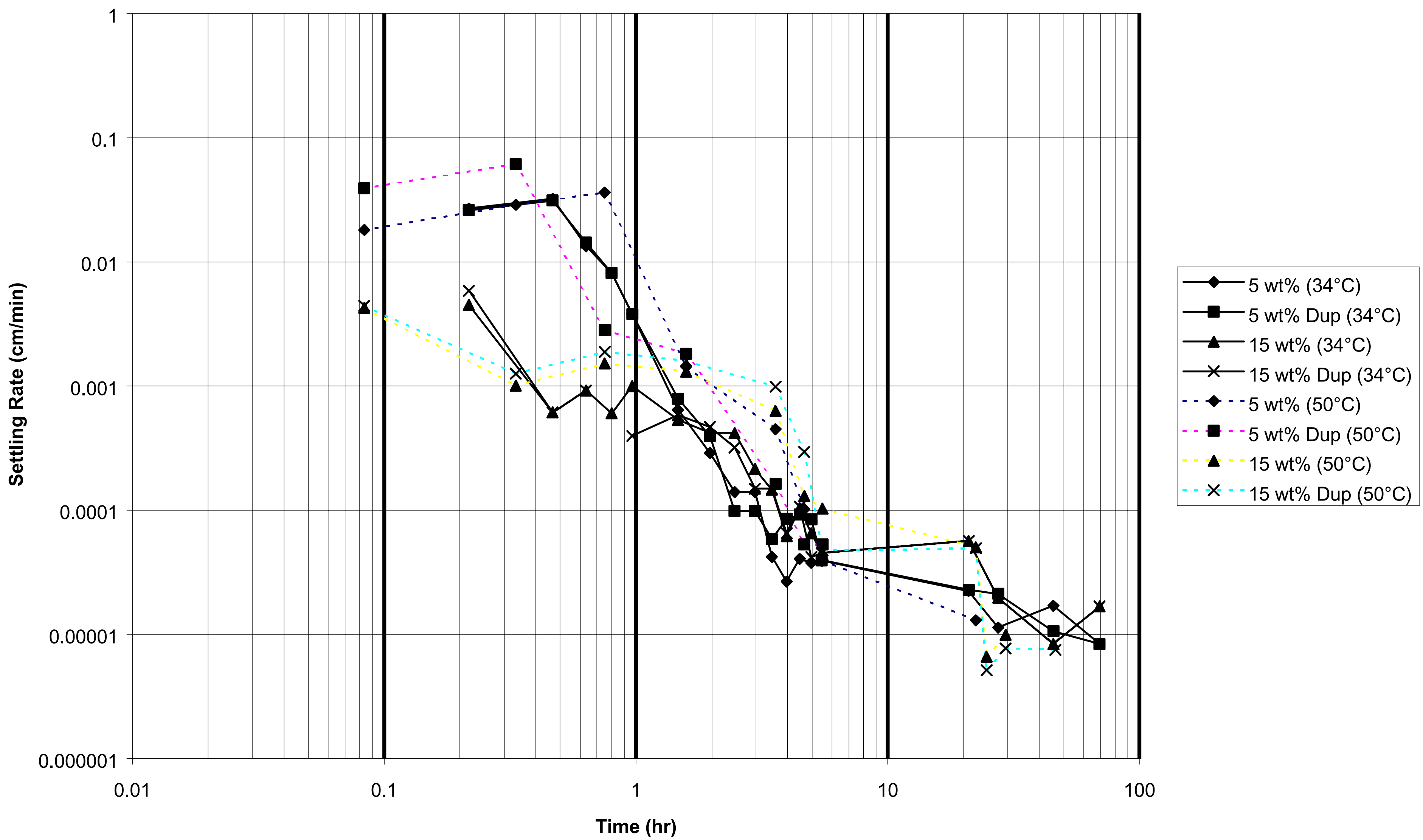


Figure 3.12. Settling Rates for C-104 Evaporated Feed With Secondary Waste Products

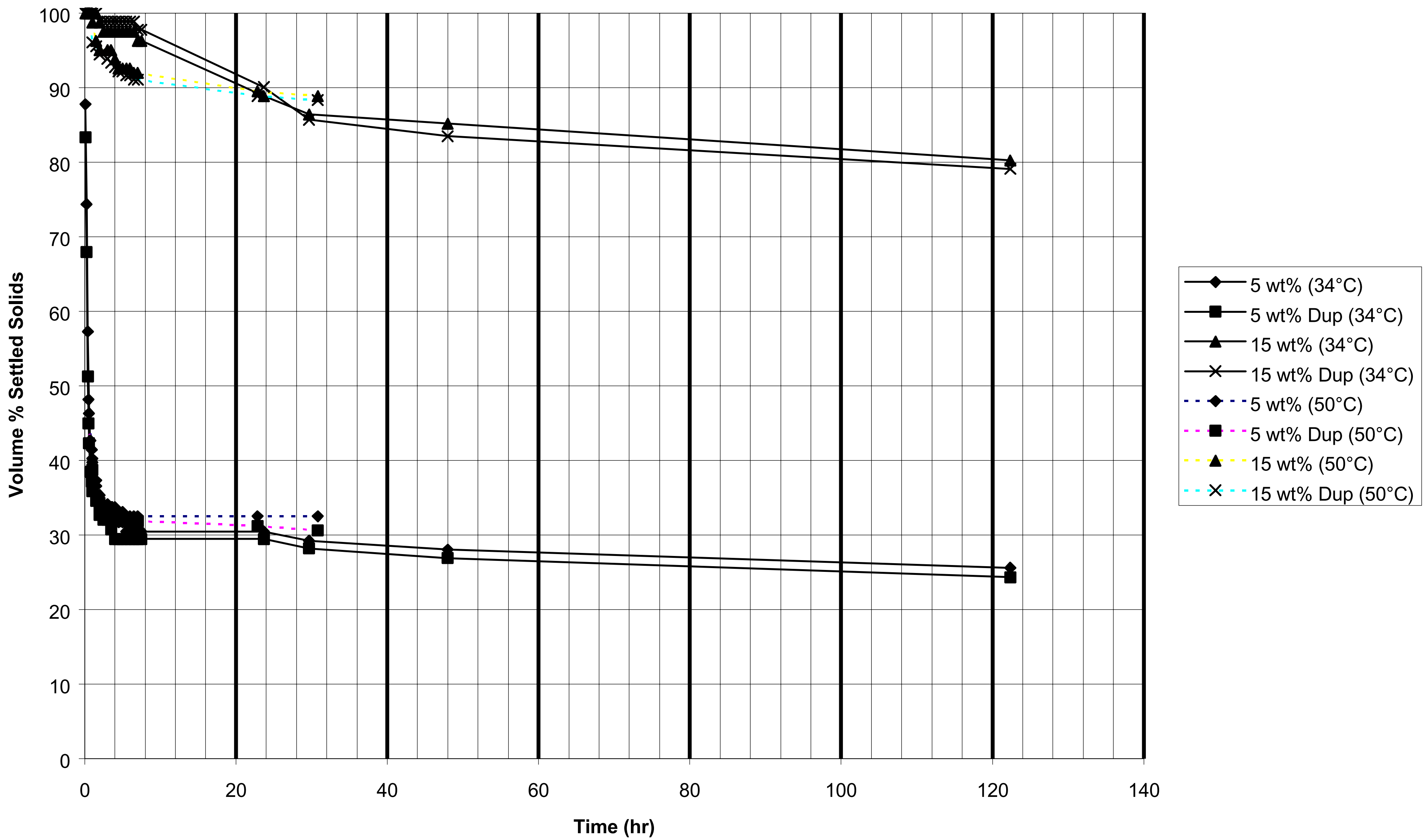


Figure 3.13. Volume Percent Settled Solids Versus Time for C-104 Evaporated Feed With Secondary Waste and Glass Formers Using a Linear Scale

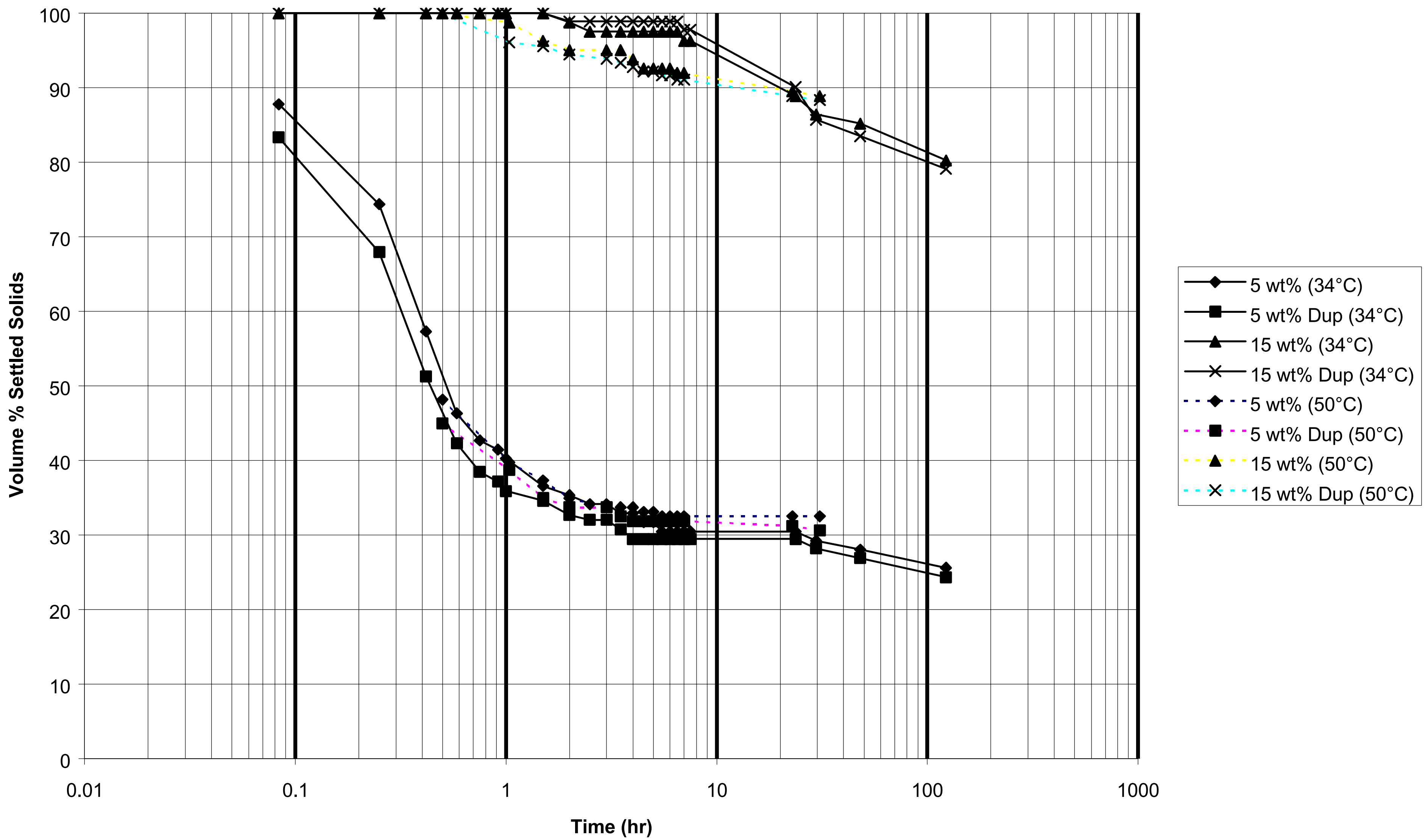


Figure 3.14. Volume Percent Settled Solids Versus Time for C-104 Evaporated Feed With Secondary Waste and Glass Formers Using a Semi-Log Scale

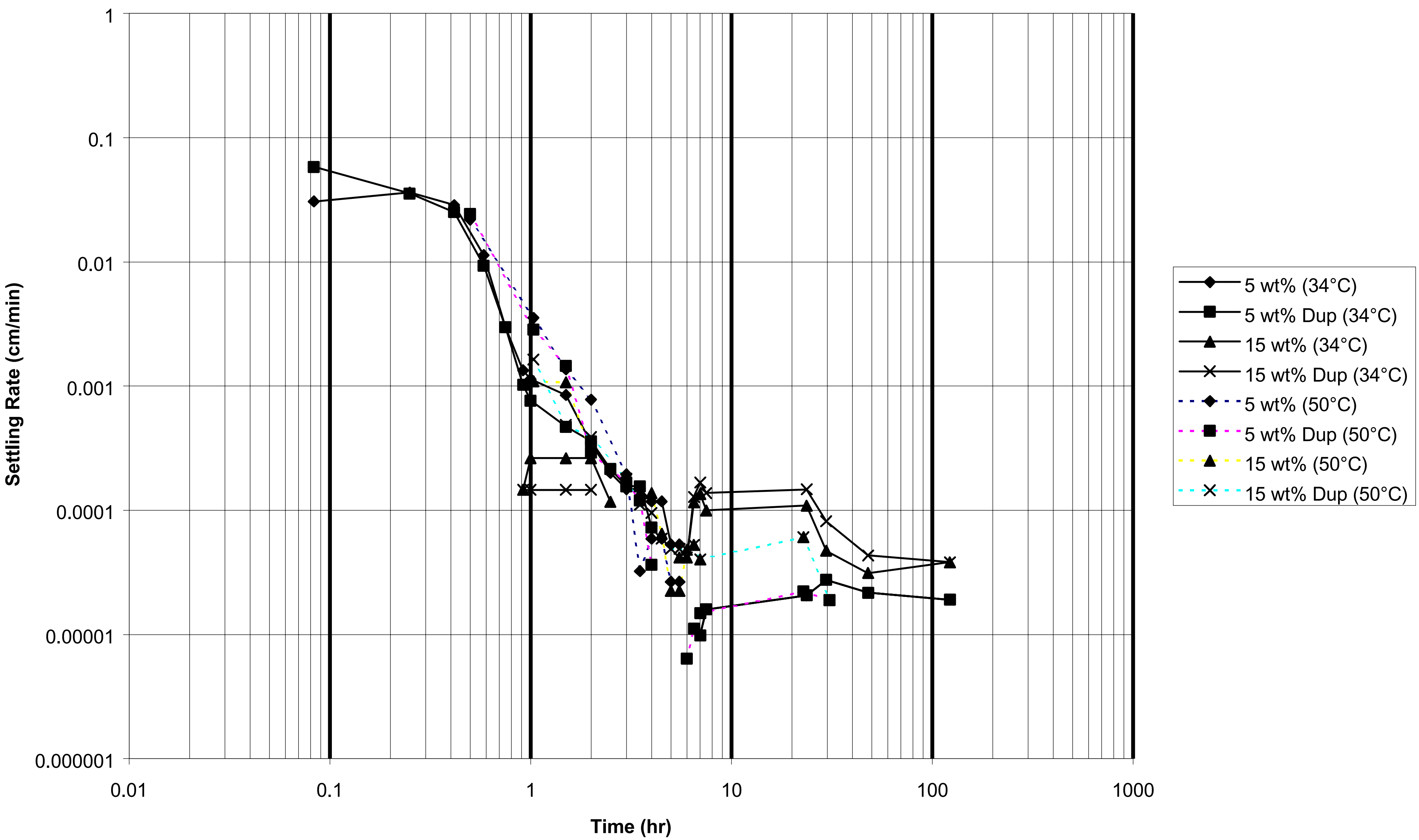


Figure 3.15. Settling Rates for C-104 Evaporated Feed With Secondary Waste Products and Glass Formers

3.2 Rheology

Viscosity is the internal resistance to flow of a fluid against external forces. Viscosity is mathematically defined as the shear stress divided by the shear rate. For a Newtonian fluid this ratio is constant. For non-Newtonian fluids this ratio can change based on flow conditions and shear history. The rheological data most often requested and provided is a rheogram. Rheograms provide flow data over a range of conditions rather than at one point. A rheometer ramps up the shear rate to chosen value while measuring and recording the resulting shear stress. From a rheogram viscosity data, yield stress data and flow curve information are obtained. There are several types flow curves that have been well studied and have defined mathematical curve fits assigned to them. These curve fits are usually used to describe and predict flow behaviors of fluids. The three that best describe most slurries and consequently tank waste are as follows:

Newtonian Equation
Tau = ETA*D

Ostwald Equation (pseudoplastic or power law fluid)
Tau = A*D^n

Herschel-Bulkley Equation (yield pseudoplastic)
Tau-Tau(o) = A*D^n

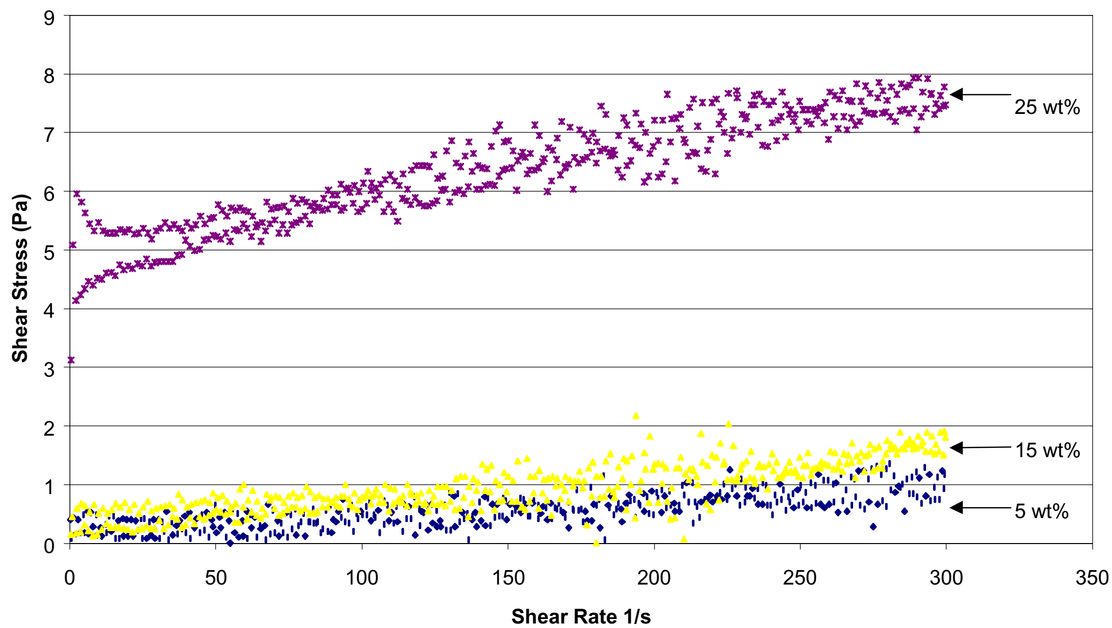
Tau = Shear Stress
ETA = viscosity
D = Shear Rate
n = power law factor n=1 fluid is Newtonian
n>1 fluid is dilatant
n<1 fluid is pseudoplastic
A = a coefficient related to flow resistance, similar to apparent viscosity. For Newtonian fluids A=viscosity.

For the C-104 and AZ-102 materials, a standard 0-300 S^{-1} ramp over 2 minutes was used to collect the increasing shear rate curve immediately followed by a 300-0 S^{-1} ramp down over 2 minutes to collect the decreasing shear rate curve. Each sample was run at least twice. The pertinent resulting rheograms and viscosity curves are included in either this section or the appendix.

3.2.1 Rheology of Evaporated Feed Samples

C-104 Feed
Shear stress vs. Shear rate curves were obtained for the C-104 waste feed at 5, 15, and 25 wt% concentrations at both 25°C and 50°C.

Rheograms for the feeds at 25°C are presented in Figure 3.16. Yield stresses and viscosities at 33, 150, and 298 S^{-1} are presented in Table 3.3. The viscosity of the 5 and 15 wt% samples was low, 15 cP or less at 25°C. The material showed predominately Newtonian behavior, with only a slight indication any yield or pseudo-plastic behavior, usually exhibited only during the first analysis. This slight non-newtonianism is most likely due to the fact that at these low viscosities the material being tested is below the recommend range for SV1 sensor utilized to obtain the data. And is therefore probably an artifact of loading stresses and initial startup machine inertia.



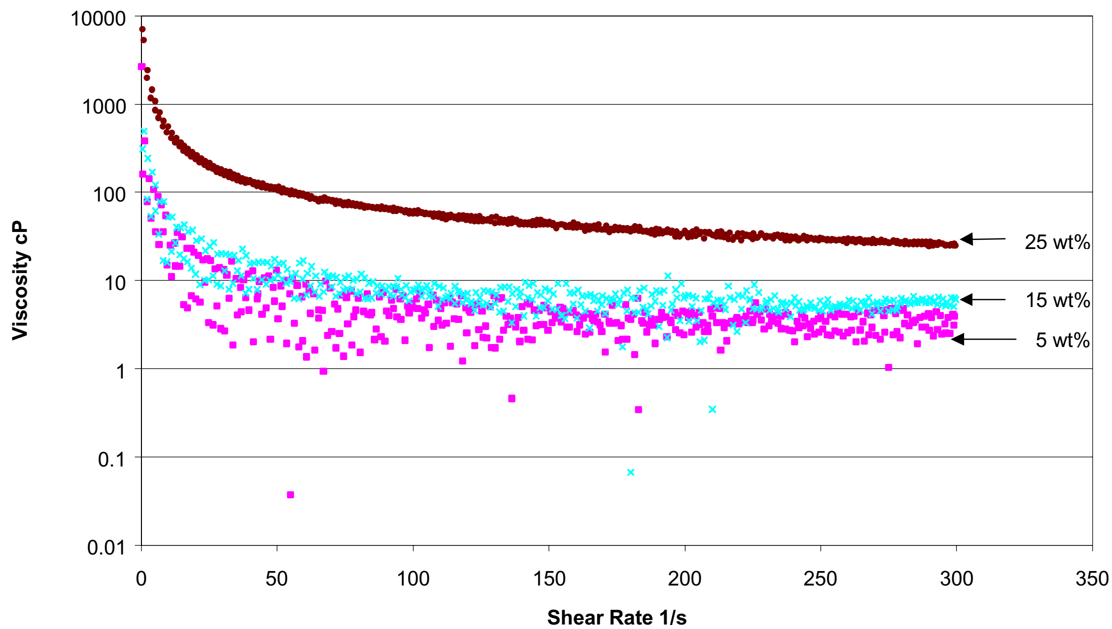


Figure 3.16. Rheograms for C-104 Initial Evaporated Feeds at 25°C

Table 3.3. Viscosity Data for Evaporated Feeds

	25°C				50°C			
Material	**Yield[a](Pa)**	**33 s^{-1}[b]**	**150 s^{-1}[b]**	**298 s^{-1}[b]**	**Yield[a](Pa)**	**33 S^{-1}[b]**	**150 s^{-1}[b]**	**298 s^{-1}[b]**
C-104 5 wt%	<1	9.8	5.2	4.7	<1	3.8	4.1	3.2
C-104 15 wt%	<1	15.4	6.2	5.7	<1	14.6	5.4	4.9
C-104 25 wt%	~5	164.9	45.5	25.9	~8	193.8	49.2	26.7
AZ-102 5 wt%	<1	12.1	5.7	5.2	<1	11.7	4.7	4.3
AZ-102 15 wt%	~14	525.2	144.1	78.7	~17	556.2	148.3	81.3
AZ-102 20 wt%[c]	~20	903.8	233.1	128.6	NA	NA	NA	NA
AZ-102 25 wt%	~190	4597.2	1263.5	702.6	NA	NA	NA	NA

a. Yield data is in Pa and is based on visual estimates from the graph and/or various curve fits. When there is a disparity between multiple runs the highest yield is reported 1 Pa was the limit for quantification.
b. Viscosity data is in cP and is an average of multiple runs at each shear rate given.
c. AZ-102 20 wt% data was obtained with new sample from material originally set aside for the mixing study.

The 25 wt% material showed a small yield stress with little pseudo-plastic behavior over the rest of the testing range. It most closely resembles a Bingham Plastic material.

Temperature Effects: The 5 and 15 wt% feeds exhibited large amounts of scatter in the data resulting from use of the SV1 sensor. No temperature effects were measurable within the range of this system.

The 25 wt% material showed an unexpected small rise in viscosity with the increased temperature. This rise is well within the range of error for this system with a change of sample, however since it was consistent it may be a real effect. This effect could also be a result of a small amount of dehydration during testing, even though visually the material did not appear to have dried out. If more material had been available a second sample would have been run at each temperature.

AZ-102 Feed

Shear stress vs. Shear rate curves were obtained for the AZ-102 waste feed at 5, 15, 20, and 25 wt% concentrations at 25°C. Yield stresses and viscosities at 33, 150, and 298 $S^{-1}$are presented in Table 3.3. Due to limited amounts of sample, only data for 5 and 15 wt% was obtained at 50°C.

The original test plan did not call for a 20 wt% measurement, however since the apparent yield and viscosity for the 25 wt% material greatly exceeded design values for processing, a 20 wt% feed was also tested. In the process of acquiring these data an important discovery was made concerning the nature of AZ-102 feed that may have later played a role in the mixing study. The AZ-102 feed appears to be irreversibly shear sensitive. The longer it is sheared or mixed the lower the apparent viscosity and yield stress may become. This was determined when the 25 wt% material was diluted down to 20 wt% and tested. The 20 wt% material showed a lower apparent viscosity and yield than the 15 wt% material. Since this was counter intuitive, the measurement was repeated two days later with a fresh aliquot from the same 20 wt% bulk sample. This resulted in even lower values. Since the material from the viscometer is returned to the bulk sample after each measurement, the bulk sample gains an increasing shear history. An untouched bottle of AZ-102 feed had been set aside for the mixing study, it was utilized as a source for fresh sample. The 20 wt% material data obtained from fresh material fell within the expected range of yield and viscosity. Thus it is most likely that the previous testing on the material originally used for the 20 wt% testing was the cause of the anomaly. This leads to the hypothesis that the AZ-102 feed may be irreversibly shear sensitive. This property could be an asset to processing. More testing would need to be done to validate this observation.

Rheograms results for the AZ-102 feeds at 25°C are presented in Figure 3.17 and does not include the highly sheared 20 wt% sample. The rheograms for this sample are in Appendix B.

The viscosity of the 5 wt% sample was mostly Newtonian in behavior, with only a slight indication any yield or pseudo-plastic behavior. This slight non-Newtonian behavior is most likely due to the fact that at low viscosities the material being tested is below the recommended range for the SV1sensor. Therefore the non-Newtonian behavior is possibly an artifact of loading stresses and inertia associated with instrument start up.

The 15, 20 and 25 wt% material displayed characteristic pseudoplastic flow behavior, each with an apparent yield stress. The data most often fit one to three of the classic curves, presented in Section 3.2, with an R^2 of 0.8-1.0. As would be expected, the yield stress and apparent viscosity increased with solids loading in a nonlinear fashion.

Temperature Effects: The 5 wt% feed exhibited large amounts of scatter in the data resulting from use of the SV1 sensor. No temperature effects were measurable within the range of this system.

As observed for the C-104 feeds, the C-104 15 wt% material showed an unexpected small rise in apparent viscosity and yield with the increased temperature. This rise is well within the range of error for this system with a change of sample, however since it was consistent it may be a real effect. This effect could also be a result of a small amount of dehydration during testing, even though visually the material did not appear to have dried out. If more material had been available a second sample would have been run at each temperature.

3.2.2 Rheology of Melter Feed with Secondary Waste Products

C-104

Rheology data was gathered after adding secondary waste products to the C-104 5, 15 and 25 wt% slurries. Yield stress and viscosities at 33, 150, and 298 S^{-1} 2 are presented in Table 3.4. Comparing Table 3.3 and 3.4, one notes there was little change between the initial evaporated feed and the feed after addition of secondary waste. The only slight change was observed in the apparent yields of the 15 wt% samples. These yields are slightly higher after adding the secondary waste. Temperature seems to have no additional effect on the C-104 sample with secondary waste.

Table 3.4. Viscosity Data (cP) for C-104 with Secondary Waste

	25°C				50°C			
Material	**Yield[a](Pa)**	**33 s^{-1}[b]**	**150 s^{-1}[b]**	**298 s^{-1}[b]**	**Yield[a](Pa)**	**33 s^{-1}[b]**	**150 s^{-1}[b]**	**298 s^{-1}[b]**
C-104 5 wt%	<1	9.6	3.5	3.4	<1	7.6	3.8	2.5
C-104 15 wt%	~2	28.7	8.0	5.6	~2.5	28.4	7.5	5.8
C-104 25 wt%	~5	158.5	44.5	27.0	~8	193.5	56.7	30.1

a. Yield data is in Pa and is based on visual estimates from the graph and/or various curve fits. When there is a disparity between multiple runs the highest yield is reported 1 Pa was the limit for quantification.

b. Viscosity data is in cP and is an average of multiple runs at each shear rate given.

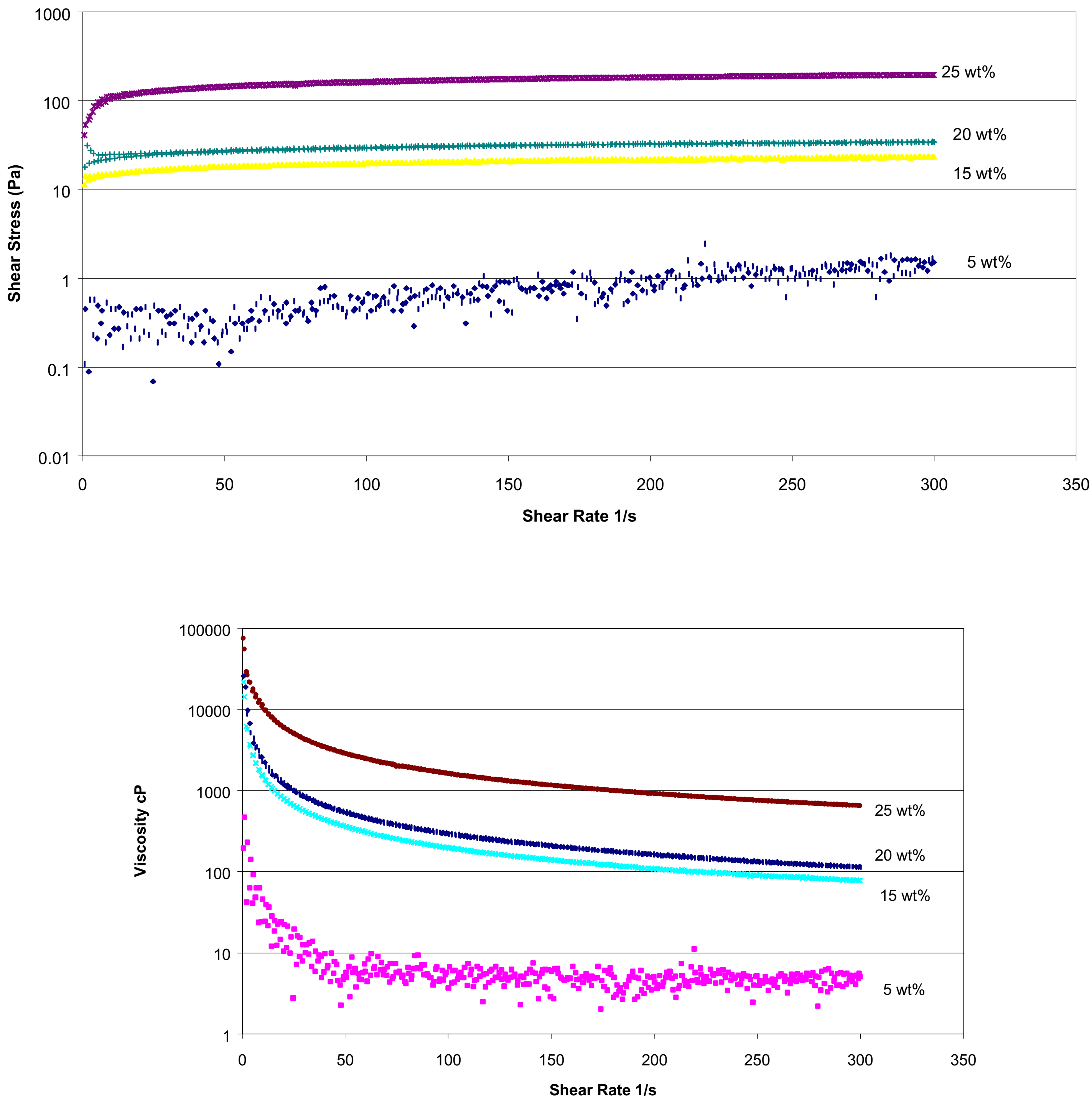


Figure 3.17. Rheograms for AZ-102 Initial Feeds at 25°C

AZ-102
As planned in Addendum 1 to Test Plan 29953-073, rheology measurements on the AZ-102 material with secondary waste products were not completed because the small amount of secondary waste products added was not expected to impact the rheology.

3.2.3 Rheology of Melter Feed with Glass Formers

C-104
The C-104 5 wt% waste shows little change with the addition of secondary waste and glass formers. Yield stress and viscosities are presented in Table 3.5. All the 5 wt% data is within the range of scatter for low viscosity materials using the SV1 sensor. The C-104 15 and 25 wt% wastes increased in both apparent yield stress and viscosity once the glass formers were added. As seen in Figure 3.18, most significant increase occurred in the 25 wt% slurry where the yield stress jumped from ~5 to ~30 Pa. These data should be considered carefully if 30 Pa is near the range of potential process constraints. Again there is the slight increase in yield stress and viscosity at the 50°C temperature range. The slurry still displays yield pseudoplastic behavior after the addition of glass formers.

Table 3.5. Viscosity Data for Evaporated Feeds with Secondary Waste and Glass Formers

	25°C				50°C			
Material	**Yield[a](Pa)**	**33 s^{-1b}**	**150 s^{-1b}**	**298 s^{-1b}**	**Yield[a](Pa)**	**33 s^{-1b}**	**150 s^{-1b}**	**298 s^{-1b}**
C-104 5 wt%	<1	8.2	4.2	4.2	<1	9.0	2.0	3.1
C-104 15 wt%	~4	138.3	45.6	29.2	~5	195.4	53.0	32.6
C-104 25 wt%	~25-30	835.2	221.7	126.1	~30-33	1075.8	265.1	146.0
AZ-102 5 wt%	<1	11.2	4.6	7.3	<1	9.2	3.8	5.0
AZ-102 15 wt%	~5	180.6	63.6	42.2	~6-7	227.0	73.4	47.7

a. Yield data is in Pa and is based on visual estimates from the graph and/or various curve fits. When there is a disparity between multiple runs the highest yield is reported 1 Pa was the limit for quantification.
b. Viscosity data is in cP and is an average of multiple runs at each shear rate given.

AZ-102
The AZ-102 5 wt% slurry maintained or slightly dropped in viscosity with the addition of the secondary wastes and glass formers, again the change was well within the error band of the equipment. However the 15 wt% slurry experienced a significant, around 50% decrease in both yield stress and viscosity as seen in Figure 3.19. Since glass formers contain no liquid, glass former addition should have resulted in an increased viscosity (although addition of certain solids could decrease viscosity). It was noted in the AZ-102 feed discussion, that it is highly probable that this material is irreversibly shear thinning, the observed decrease is probably the result increased shear history and not addition of glass formers alone.

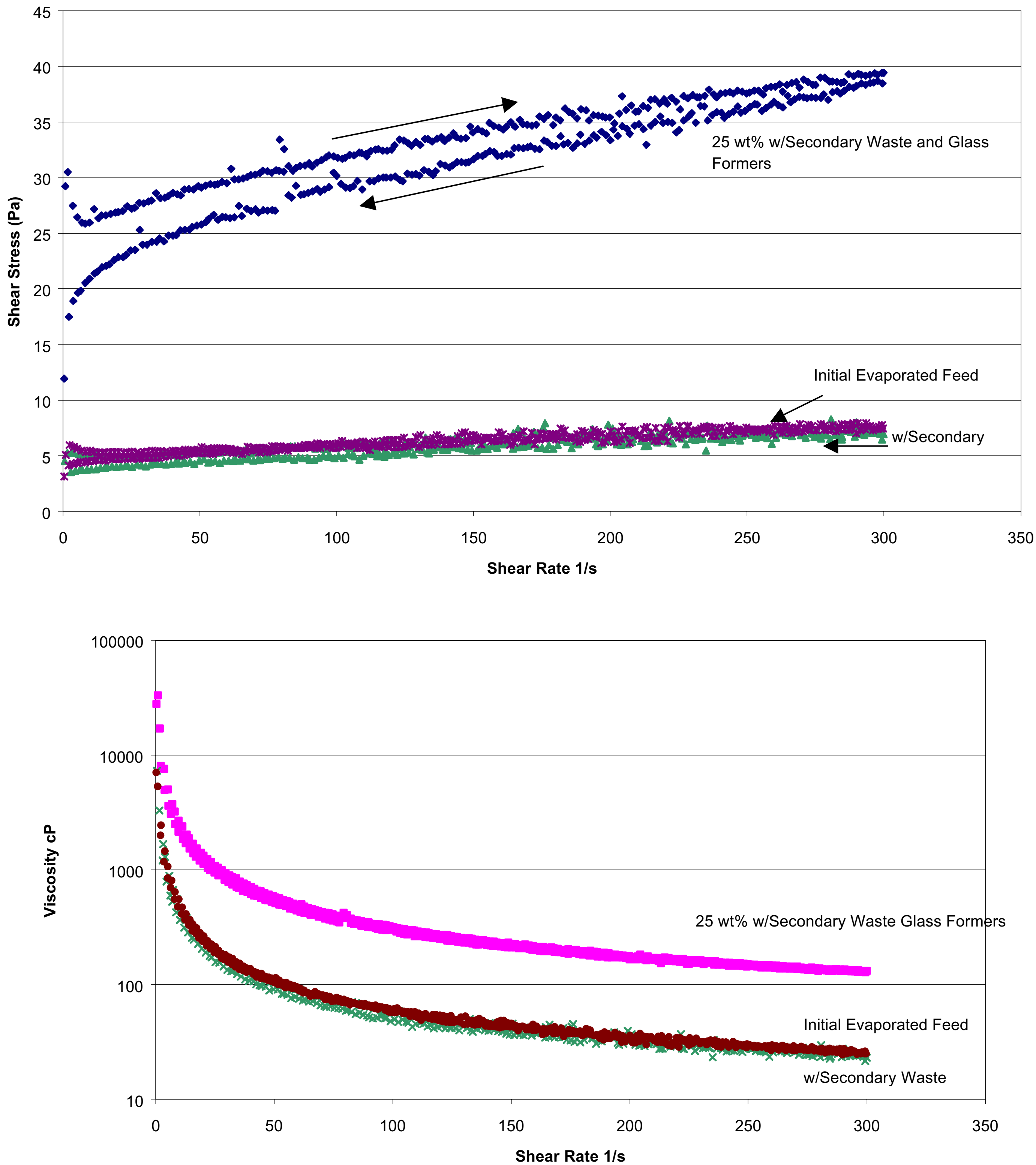


Figure 3.18. Comparison of C-104 25 wt% Feed Initial Alone, With Secondary Waste and With Glass Formers and Secondary Waste at 25°C

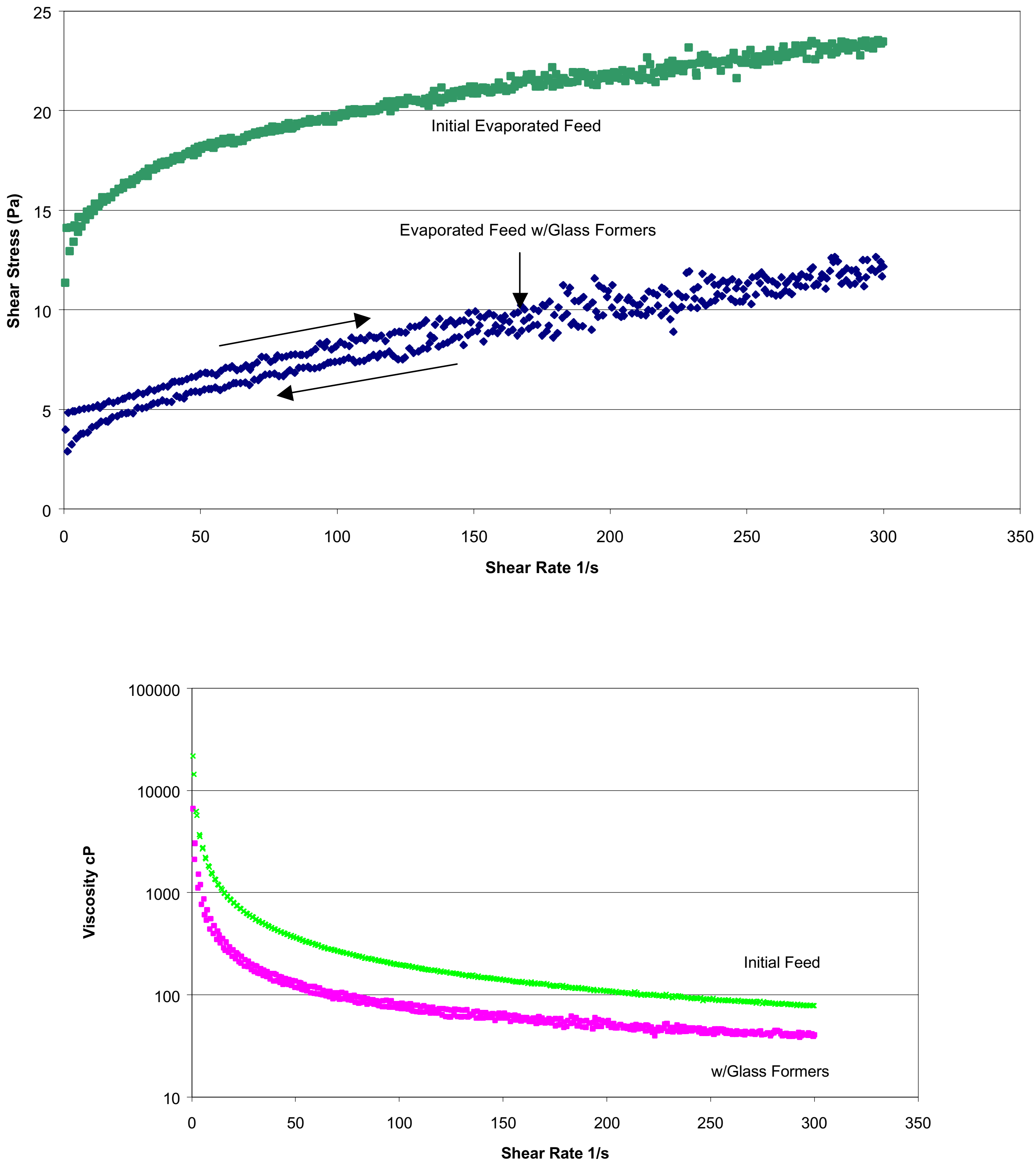


Figure 3.19. Comparison of AZ-102 15 wt% Feed Alone and With Glass Former at 25°C

3.3 AZ-102 Mixing and Aging Study

A fresh sample of AZ-102 15 wt% solids, with both secondary wastes and glass formers added, was mixed for over for a week at ambient temperature (34°C). Rheology measurements were taken at 1 hour, 1 day and 1 week at 25°C. The AZ-102 slurry decreased significantly in both yield stress and viscosity with time while mixing as seen in Figure 3.20. This decrease with mixing fits well within the irreversible shear thinning observed previously for the AZ-102 material.

The material was then rinsed into a graduated bottle and the excess water added to it in the transfer process was evaporated off. The slurry was then allowed to settle for 1 week at ambient temperature (34°C) and observed for gas retention and release. No gas retention or release was observed. At this point the test plan called for any excess liquid to be drained and a rheology measurement made of the settled solids. The standing liquid was removed from the AZ-102 and the solids were measured. Rheograms are presented in Figure 3.21. Yield stress and viscosity data are presented in Table 3.6.

One will note a drastic increase in the viscosity and yield after settling for one week. While this seems to contradict irreversible shear thinning, one must remember this data was collected on the settled solids following removal of standing liquid. There was no standing liquid on the initial 15 wt% evaporated feed, so standing liquid on the same feed after addition of dry glass formers shows a significant change in solids structure.

Table 3.6. Viscosity Data for AZ-102 Mixing and Aging Study Measured at 25°C

Material	**Yield[a](Pa)**	**33 s^{-1}[b]**	**150 s^{-1}[b]**	**298 s^{-1}[b]**
AZ-102 1 hr	~6-7	252.5	80.3	49.5
AZ-102 1 day	~3	123.5	41.5	27.1
AZ-102 1 week	~1	51.9	18.7	14.8
AZ-102 Settled 1 week	~6-12	276.6	92.2	58.7

a. Yield stress data is in Pa and is based on visual estimates from the graph and/or various curve fits. When there is a disparity between multiple runs the highest yield is reported. The limit for quantification was 1 Pa.

b. Viscosity data is in cP and is an average of multiple runs at each shear rate given.

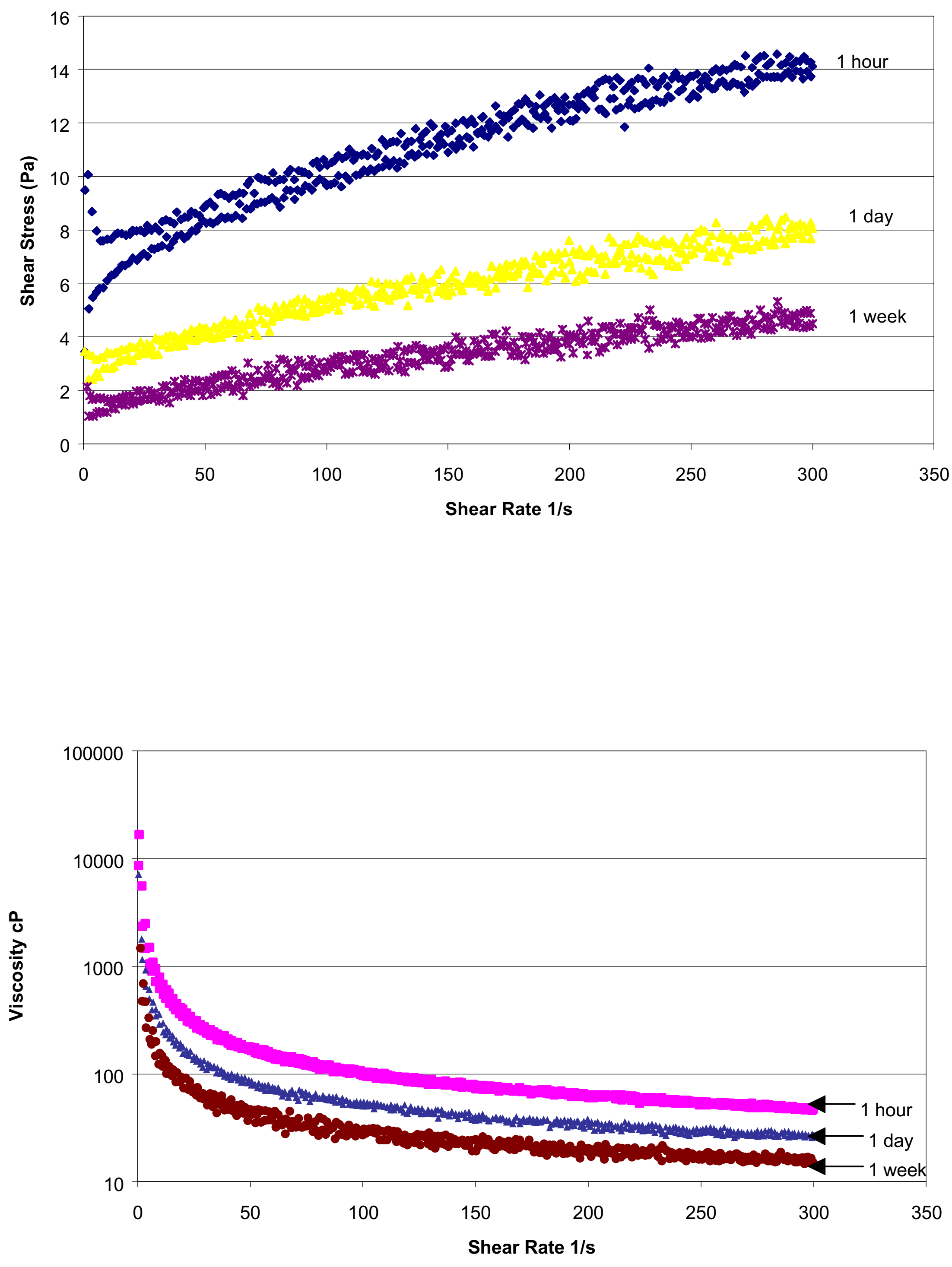
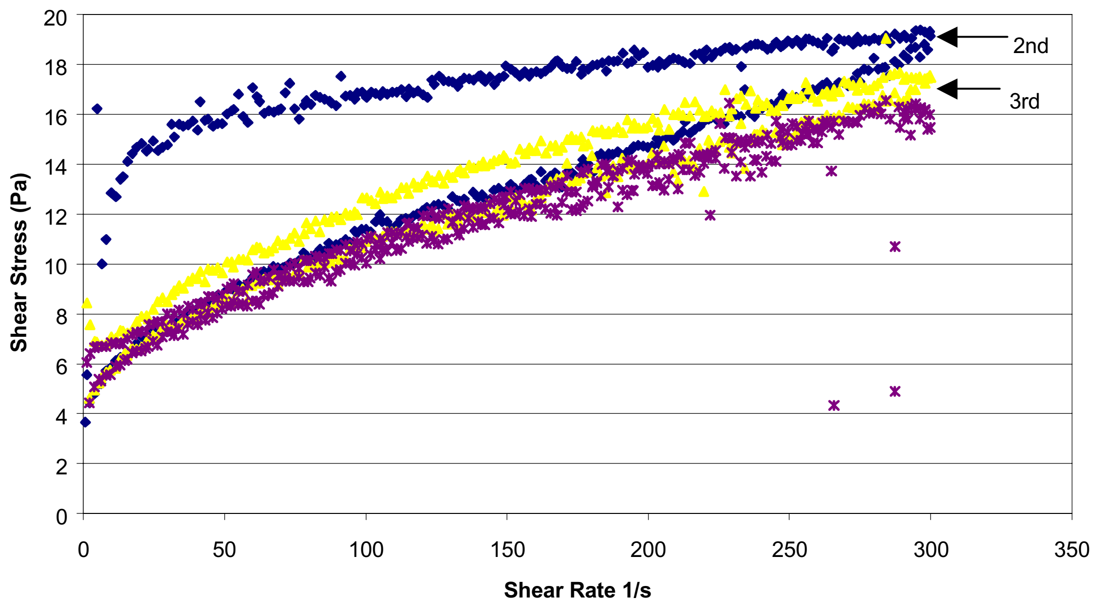


Figure 3.20. Rheograms for AZ-102 15 wt% Mixing Study, Measurement Performed at 25°C



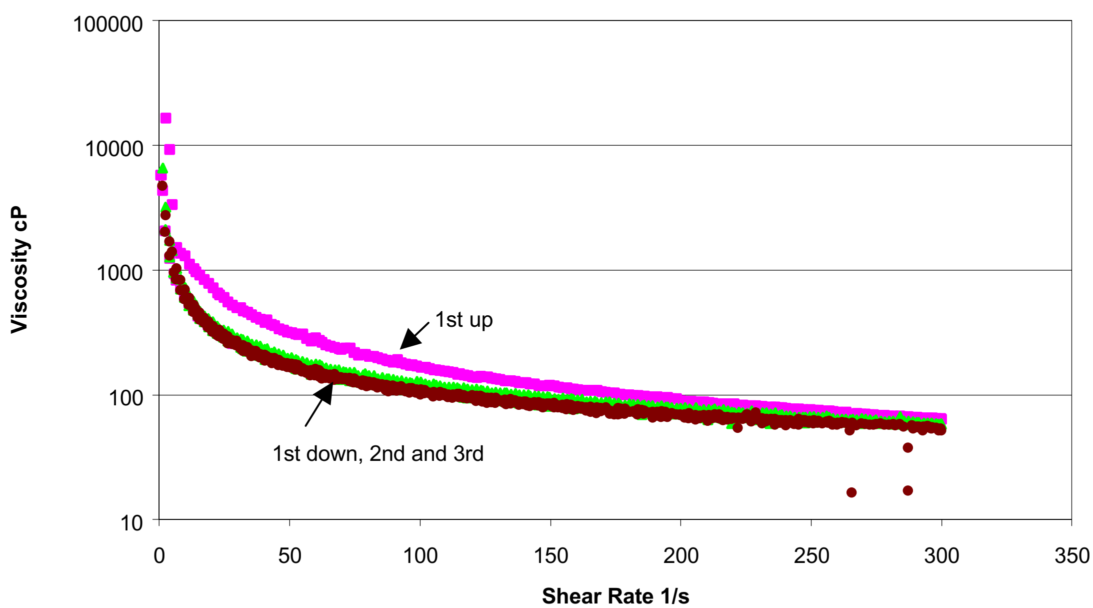


Figure 3.21. Rheograms for AZ-102 15 wt% Aging Study Measurements Performed at 25°C

3.4 C-104 Mixing and Aging Study

A fresh sample of C-104 25 wt% solids with both secondary wastes and glass formers added was mixed for a week at ambient temperature (34°C). Rheology measurements were taken at 1 hour, 1 day and 1 week at 25°C. Yield stress and viscosity data are summarized in Table 3.7. As seen in Figure 3.22, the C-104 slurry increased in both yield stress and viscosity with time while mixing. The material started out with a yield of ~28 Pa which is already close to many processing/design limitations and climbed to over 55 Pa by the end of the week.

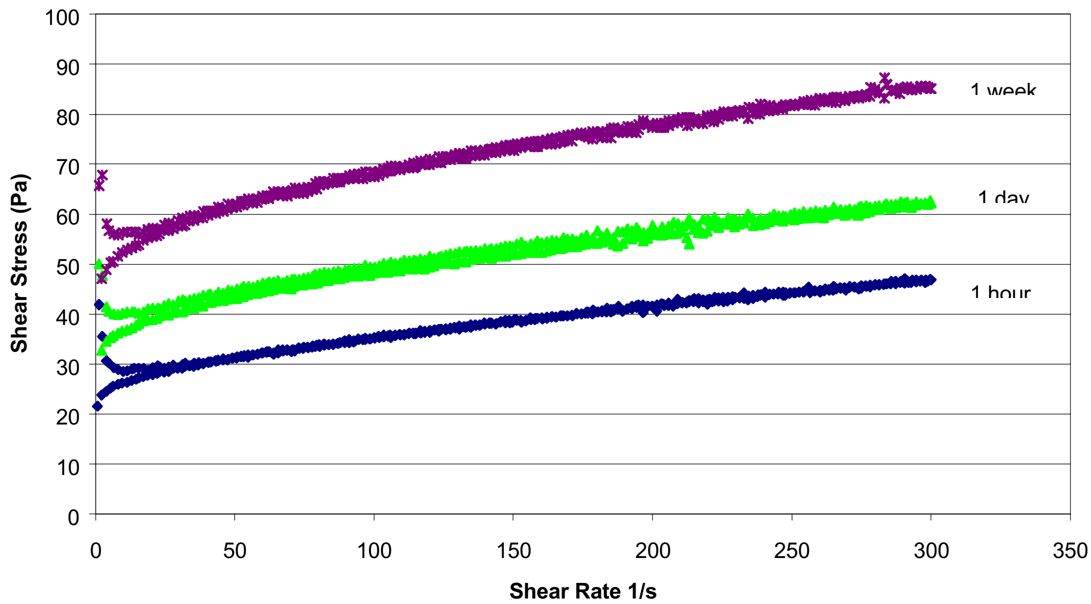
The material was then rinsed into a graduated bottle and the excess water added in the transfer process was evaporated off. The slurry was then allowed to settle for 1 week at ambient temperature (34°C) and observed for any gas retention and release. No gas retention or release was observed. At this point the test plan called for any excess liquid to be drained and a rheology measurement made of the settled solids at 25°C. There was no extra liquid to remove from the C-104 material after the 1-week aging period. As seen in Figure 3.23, yield stress of the aged settled solids spiked at over 80 Pa before settling down to ~40 Pa on repeat analysis. Materials with a yield stress this high could be difficult to pump and process.

Table 3.7. Viscosity Data for C-104 Mixing and Aging Study Measured at 25°C

Material	**Yield[a](Pa)**	**33 S^{-1}[b]**	**150 S^{-1}[b]**	**298 S^{-1}[b]**
C-104 1 hr	~28	914.5	258.9	157.2
C-104 1 day	~38	1264.5	351.3	209.1
C-104 1 week	~56	1740.5	479.7	282.3
C-104 Aged 1 week	~ 80	1621.3	421.8	240.7

a. Yield data is in Pa and is based on visual estimates from the graph and/or various curve fits. When there is a disparity between multiple runs the highest yield is reported. The limit for quantification was 1 Pa.

b. Viscosity data is in cP and is an average of multiple runs at each shear rate given.



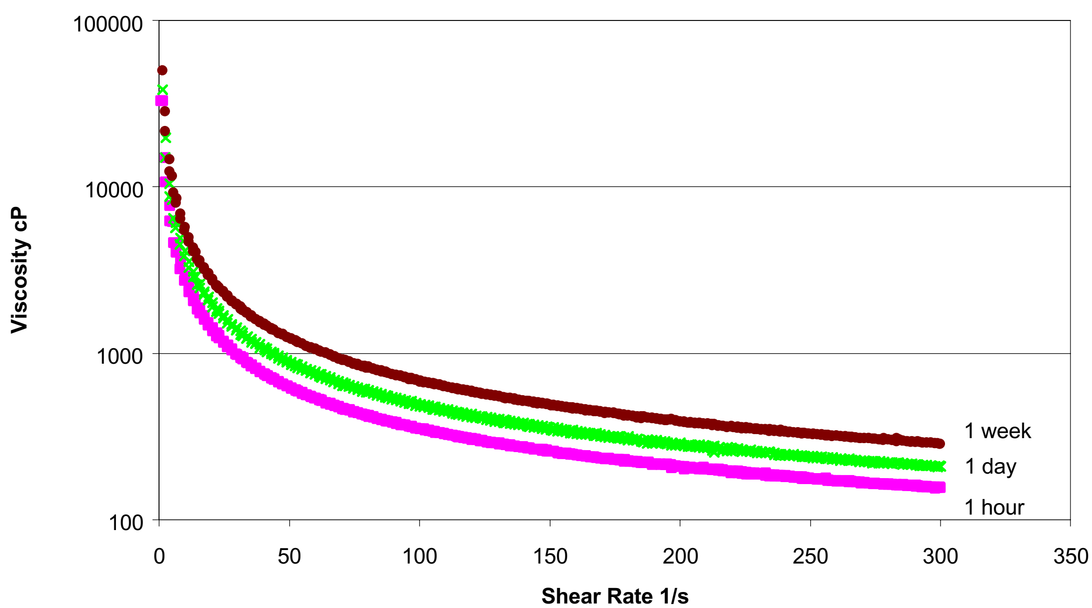
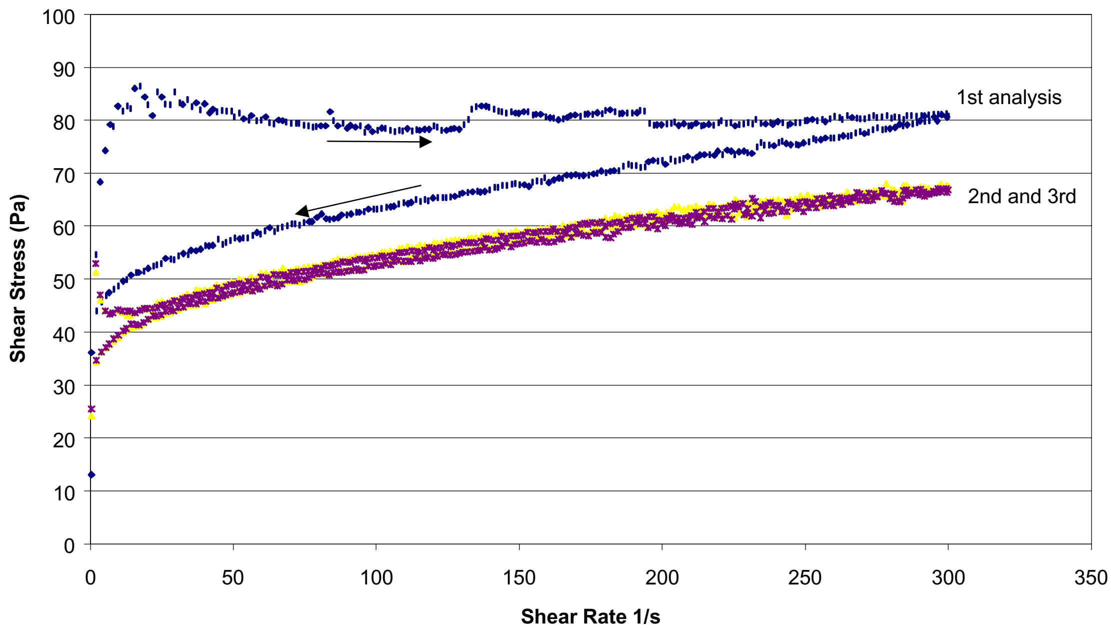


Figure 3.22. Rheograms for C-104 15 wt% Mixing Study Measurement Performed at 25°C



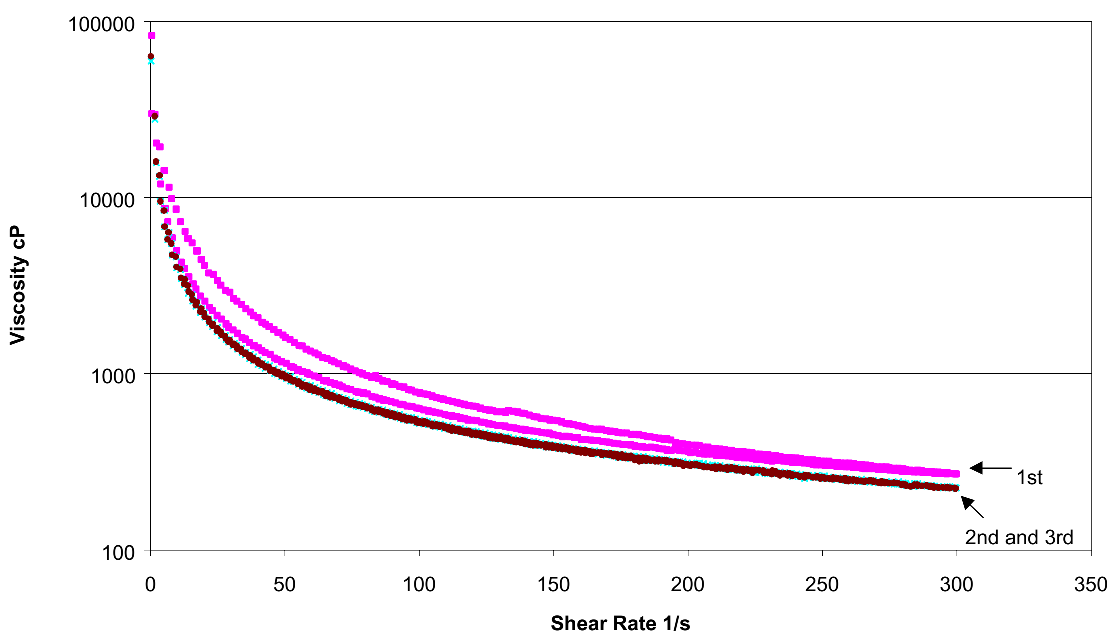


Figure 3.23. Rheograms for C-104 25 wt% Aging Study Measurement Performed at 25°C

4.0 Conclusions

The following conclusions were made based on the rheology and physical properties of the AZ-102 and C-104 evaporator and melter feeds. These conclusions have been divided into categories for clarity.

Concentration/Evaporation

- The AZ-102 initial feed was received at 9.54 wt% solids with no standing liquid. Therefore, concentration of the feed to 15 and 25 wt% resulted in thick slurries with no standing liquid.

- The C-104 initial feed was received at 20 wt% solids with roughly 15 vol% standing liquid. Evaporation of this feed to 25 wt% resulted in a slurry with little to no standing liquid after extended settling periods of greater than 3 days.

Density and Settling Behavior

- As expected, the densities of the samples increased with solids concentration ($\rho_{25wt\%}>\rho_{15wt\%}>\rho_{5wt\%}$) and increased following the addition of secondary waste products and increased again after the addition of glass formers.

- No significant temperature trends were observed in sample densities between 34 and 50°C. This is attributed to a lack of soluble solids.

- Samples did not compact as effectively at 50°C as at 34°C. This increase in settled solids volume ranged between 4 and 8 vol%. This is probably the result of the test methodology breaking up the aggregate structure during sample agitation between the 34°C test and the 50°C test.

- Addition of secondary waste products (primarily Sr/TRU precipitate) to the C-104 evaporated feeds increased the initial solids settling rate by an order of magnitude. This is most notable in the 5 wt% feed supernatant, which remained cloudy after 3 days of settling. However, after secondary waste product addition, this sample settled with a clarified supernatant. This behavior is most likely the result of flocculation of the sludge particles with the Sr/TRU secondary waste product.

- Addition of glass formers to the AZ-102 samples appears to have increased the settling rate by approximately a factor of four. However, the increase in settling rate for the AZ-102 samples in much less dramatic than the increase for the C-104 samples.

Rheology of Evaporated Feeds

- No significant temperature effects were seen for the C-104 or AZ-102 samples measured at 25 and 50°C.

- The AZ-102 feed displayed initial viscosities of 12, 530, 900, and 4600 cP for the 5, 15, 20, and 25 wt% feeds. In addition, the AZ-102 feeds displayed significant irreversible shear thinning.

Rheology with Secondary Waste Products and Glass Formers

- Very little change in rheological properties were observed following secondary waste products (Sr/TRU precipitate and Cs eluants) and glass former addition to the C-104 feed.

- The yield and viscosity the AZ-102 feed dropped following glass former and secondary waste addition (Sr/TRU precipitate). The observed decrease is most likely the result of the increasing shear history of the AZ-102 samples as the testing progressed.

Melter Feed Mixing and Aging

- The yield stress and viscosity of the AZ-102 slurry decreased over the one week mixing period consistent with the previous observations in this report of irreversible shear thinning in this slurry.

- Following one week of settling, the yield stress and viscosity of the AZ-102 settled solids were higher than similar testing after one week of mixing. This increase was expected and attributed to increased solids loading resulting from removal of the standing liquid from the aged sample.

- The results of the mixing and settling studies suggest that while the AZ-102 initial feed and final melter feed material may be very difficult to mix and transport, irreversible shear thinning significantly reduces the yield and viscosity. Therefore, exposing this material to some initial shearing may be an option for reducing these critical transport properties.

- The yield stress and viscosity of the C-104 slurry increased over the one week mixing period. The C-104 slurry yield stress increased from 28 Pa after one hour to 56 Pa after one week. Over this same period, the viscosity of the C-104 slurry increased from 910 cP to 1700 cP at $33s^{-1}$.

- After standing for one week, the viscosity of the C-104 solids did not settle into a separate layer, so no liquids were decanted. The viscosity of the C-104 settled material was similar to the viscosity after one week of mixing, but the yield stress increased to approximately 80Pa.

- The yield stress and viscosity of the C-104 melter feed increased with mixing and the yield increased further with aging. These results suggest the final C-104 melter feed could be very difficult to mix and transport on a 25 wt% basis.

5.0 References

Bredt, PR and RG Swoboda. 2000. "Rheological Studies on Pretreated Feed and Melter Feed From AW-101 and AN-107," PNWD-3034, Revision 0, Battelle Pacific Northwest Division, Richland, Washington.

Brooks, KP, PR Bredt, GR Golcar, SA Hartley, LK Jagoda, KG Rappe, MW Urie. 2000. "Characterization, Washing, Leaching, and Filtration of C-104 Sludge," PNWD-3024, Battelle Pacific Northwest Division, Richland, Washington.

Brooks, KP et al. 2000. "Characterization, Washing, Leaching, and Filtration of AZ-102," PNWD-3045, Battelle Pacific Northwest Division, Richland, Washington.

APPENDIX A

Appendix A: Test Plans

APPENDIX B

Appendix B: Rheograms

APPENDIX C

Appendix C: Test Instruction

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Appendix A: Test Plans

PNNL Test Plan

Document No.: BNFL-TP-29953-073
Rev. No.: 0
Document Control: Only the original signed copy is controlled

Title: High Activity Melter Feed Rheological and Physical Properties Measurements

Work Location: Radiochemical Processing Laboratory	**Page** 1 of 60
Author: Paul Bredt **Use Category Identification: Reference**	**Effective Date:** Upon Final Approval **Supersedes Date: New**
Identified Hazards: ___ Radiological ___ Hazardous Materials ___ Physical Hazards ___ Hazardous Environment ___ Other:	**Required Reviewers:** X Technical Reviewer X Building Manager __ Radiological Control __ ES&H X Quality Engineer X Project Manager X RPL Manager __ SFO Manager

Are One-Time Modifications Allowed to this Procedure? X Yes __ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? _____ Yes or X No

FOR REVISIONS:
Is retraining to this procedure required? _____Yes X No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
_____Yes _____No X N/A

Approval	**Signature**	**Date**
Author	Paul Bredt	12/6/99
Technical Reviewer	Gary L. Smith	12/20/99
RPL Manager	LO Casazza	12/16/99
Project Manager	Eugene Morrey	1/20/00
Building Manager	Rud [illegible]	1-21-00
RPL QE	Taffy L. Almeida	1-21-00
BNFL	S [illegible]	1/21/00

High Activity Melter Feed Rheological and Physical Properties Measurements

Scope

Physical and rheological properties will be measured for the waste sample from tanks AZ-102 and C-104. Solids settling rate, density and shear stress versus shear rate rheograms will be measured on the samples after concentration to three undissolved solids concentrations (typically, 5wt%, 15wt% and 25wt%, i.e. including dilution and evaporation of the pretreated sample) at ambient temperature and at 50°C. The same measurements will be conducted on the three mixtures after addition of the prescribed quantities of intermediate waste products (^{137}Cs, ^{99}Tc and ^{90}Sr/TRU) and then glass formers (typically 300 g/*l*, or 500 g/*l*). The viscosity of one slurry will also be measured after 1 hour, 1 day and 1 week; the slurry will be mixed to provide a shear rate consistent with that expected in the facility. The viscosity of this slurry will be further measured after 1 week with no mixing.

This document satisfies the requirements of a test instructions which provides specific details for the implementation of Technical Procedure 29953-010, "Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges". Client expectations for successful achievement of project data needs have already been established via the "HLW Melter Feed Rheology" test specifications, numbered **TS-W375LV-TE00002 and TS-W375HV-TE0006,** provided to Battelle by BNFL.

Justification of Use Category

This procedure is reference use. Reference use was selected as the use category since this analysis is not a complex process and there are no safety impacts to the order of events. In addition, we may wish to modify the order of analyses or eliminate some analyses depending on the needs at the time of the operation.

Applicability

This test plan applies to RPL staff performing work on BNFL Privatization samples under Project 29953.

Work with actual samples is to be performed in hot cells in the RPL by staff under the direction of a cognizant scientist.

Prerequisites

1) Keep the sample in a sealed glass container as much as possible to prevent it from drying and reduce the potential for organic contamination.
2) Cross-contamination between samples and contamination of samples from outside sources must be minimized at each step. Use new tools and bottles for each sample as much as possible. Those tools which are reused should be washed and rinsed prior to reuse.
3) Secondary containment is to be used whenever practical to minimize sample loss in the event of a spilled sample or broken sample bottle.
4) This Test Plan requires work in an oven or viscometer at 50°C. Unless otherwise indicated, a value of ±5°C is acceptable.

Quality Control

Quality control has been implemented in Technical Procedure 29953-010, "Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges". Work shall be performed in accordance with the current revision of Battelle's quality assurance plan, BNFL-QAPjP.

M&TE List:

_____ Balance 1:

Calib ID 384-06-01-004 Calib Exp Date 8/2000

Location Front face HLRF

_____ Balance 2:

Calib ID 368-06-01-020 Calib Exp Date 8/00

Location c-cell

_____ Thermocouple:

Calib ID 03003 Calib Exp Date 8/00

Location A-Cell

_____ Digital Thermometer:

Calib ID PNL000 2927 Calib Exp Date 3/01

Location Front face 325 HLRF

Work Flow

1) Load 5 subsamples of a waste in graduated centrifuge tubes and place in vacuum jar.
2) Evacuate jar and monitor sample masses. To accelerate the evaporation, it may be necessary to heat the samples. Heating should be done in an oven at 50°C (±10°C).
3) At predetermined mass loss intervals, remove one centrifuge tube at a time from the jar and cap.
4) After a predetermined time interval, centrifuge the cones and measure the vol% and wt% centrifuged solids. Use this data to plot correlation between mass loss and wt% solids.
5) Load three graduated jars with same waste type.
6) Apply a vacuum to the jars and monitor the mass/volume loss until the three desired loss levels have been reached. To accelerate the evaporation, it may be necessary to heat the samples. Heating should be done in an oven at 50°C (±10°C).
7) Measure the density of the material in the graduated jars and record the ambient cell temperature.
8) Stir the jars and monitor the settled solids volumes over a 3 day period at ambient (record the ambient cell temperature.
9) Place the jars in an oven at 50°C (±5°C) for 1 day.
10) Measure the density of the material in the graduated jars at 50°C (±5°C).
11) Stir the jars and monitor the settled solids volumes over a 3 day period at 50°C (±5°C).
12) While stirring, remove subsamples from each of the three graduated jars in duplicate and analyze for shear stress versus shear rate at ambient cell temp and 50°C.
13) Add the prescribed quantities of intermediate waste products (^{137}Cs, ^{99}Tc and ^{90}Sr/TRU) and stir for one hour using an overhead mixer.
14) Repeat steps 7 through 12.
15) Add the prescribed quantities of glass formers to each of the three jars and stir for one hour using an overhead mixer.
16) Repeat steps 7 through 12.
17) Transfer one of the three samples prepared in step 15 to an airtight mixing vessel with sampling port.
18) Mix this sample for one week.
19) During the week of mixing, measure the shear stress as a function of shear rate on this samples after 1 hour, 1 day, and 1 week.
20) After the week of stirring, transfer the material back to the graduated jar and leave undisturbed for 1 week.
21) During this one week, monitor for gas retention/release behavior.
22) After one week, measure the shear stress versus shear rate on the sample.

Work Instructions

AZ-102

1) Record the solids concentration of the AZ-102 feed.

Wt% solids= 9.54% (C1) Data Source BNFL-TI-29953-079 Rev. 1 addendum 3

2) Record the Wt% solids targets provided by BNFL.

Wt% solids = 5 (C2a)

Wt% solids = 15 (C2b)

Wt% solids = 25 (C2c)

3) Load 8 ml of AZ-102 material into 5 preweighed centrifuge cones. Record the new weight.

AZ 1		AZ 2		AZ 3	
Total	____g	Total	____g	Total	____g
Cone	____g	Cone	____g	Cone	____g
Slurry	____g	Slurry	____g	Slurry	____g

AZ 4		AZ 5	
Total	____g	Total	____g
Cone	____g	Cone	____g
Slurry	____g	Slurry	____g

skipped as per Email with Stuart Arm on 3/30/00 PRB

4) Remove the caps, and place the centrifuge cones in a vacuum jar. If necessary to accelerate evaporation, place the jar in an oven at 50°C (±10°C).

5) At intervals determined by the cognizant scientist, remove the cones from the vacuum jar, replace the caps, and record the new weight.

AZ 1		AZ 2		AZ 3	
Total	____g	Total	____g	Total	____g
Cone	____g	Cone	____g	Cone	____g
Slurry	____g	Slurry	____g	Slurry	____g

AZ 4		AZ 5	
Total	____g	Total	____g
Cone	____g	Cone	____g
Slurry	____g	Slurry	____g

Attachment to BNFL-TP-29953-073
PRB 4/13/00

Bredt, Paul R

From:. Stuart Arm [sarm@bnflinc.com]
Sent: Thursday, March 30, 2000 12:46 PM
To: Bredt, Paul R
Cc: Smith, Gary
Subject: Re: AZ-102, C-104 Evap for Melter Feed Rheology Testing

Paul,
Good idea, please proceed as you describe.

Stuart.

"Bredt, Paul R" wrote:

Stuart,

Given that the supernatant associated with the AZ-102, and C-104 samples have little to dissolved solids, we were hoping to get your concurrence on a simplification of Test Plan 29953-073.

Under steps 3-7 and 66-70 we were planing on running 5 test samples for each waste to determine the amount of moisture that must be evaporated to reach the 5, 15, and 25 wt% solids levels. Instead, we would like to use the current wt% solids numbers for our basis for solids content and assume no solids will precipitated during the evaporation. We would then evaporate the samples by applying a vacuum and flowing air over the material though a pinhole in the lid.

Please let me know as soon as possible if this approach is acceptable, and we will start the evaporation (I still owe you an addendum for glass formers, but I have put it off a until next week as we are collecting more information on AZ-102 and the Sr/TRU ppt).

Thanks,
Paul

6) Centrifuge the cones at ~1000 x g for one hour. Record the solids and liquid volumes.

AZ 1		AZ 2		AZ 3	
Total	______ ml	Total	______ ml	Total	______ ml
Solid	______ ml	Solid	______ ml	Solid	______ ml
Liquid	______ ml	Liquid	______ ml	Liquid	______ ml

AZ 4		AZ 5	
Total	______ ml	Total	______ ml
Solid	______ ml	Solid	______ ml
Liquid	______ ml	Liquid	______ ml

7) Decant the centrifuged liquid to a preweighed graduated cylinder. Record the mass and volume of liquid in the graduated cylinder.

see page 5 PRB 4/3/00

AZ 1		AZ 2		AZ 3	
Total	______ g	Total	______ g	Total	______ g
Grad	______ g	Grad	______ g	Grad	______ g
Liquid	______ g	Liquid	______ g	Liquid	______ g
Liquid	______ ml	Liquid	______ ml	Liquid	______ ml

AZ 4		AZ 5	
Total	______ g	Total	______ g
Grad	______ g	Grad	______ g
Liquid	______ g	Liquid	______ g
Liquid	______ ml	Liquid	______ ml

8) Based on the results from AZ 1 through AZ 5, determine the amount of water on a wt% basis to evaporate from the three samples to reach the target solids concentrations. Attach the calculations to this Test Plan. See attached PRB 4/3/00

9) Weight three appropriately sized glass graduated jars or cylinders labeled **AZ EVAP A**, **AZ EVAP B**, and **AZ EVAP C**. Jars Labeled "AZ-102 Washed solids"

	Rheol-5 ~~AZ EVAP A~~	Rheol-15 AZ EVAP B	Rheol-25-1 ~~AZ EVAP C~~	Rheol-25-2
Tare	134.58 g	134.16 g	217.49 g	217.50 g
slurry	41.84 g	122.00 g	136.53 g	136.32 g
water ~~target~~	37.99 g	(44.41) g	(84.43) g	(84.30) g
target masses	214.41 g	211.75 g	269.59 g	269.52 g

saved for mixing & Aging Study

Battelle
Pacific Northwest Laboratories

Page ______ of ______

ENGINEERING WORKSHEET

Prepared By: PR Bredt | Date: 4/3/00 | Project: BNFL 29953

Subject: AZ-101 · Evaporation under step 8 of Test plan 29953-073

Samples were prepared (subsampled) under

BNFL-TI-29953-079 Rev. 1 addendum ~~3~~ 2 PRB

Sample solids content is 9.54 wt% under addendum 3

5 wt% sample:

Current mass = 41.84 g

$C_1M_1 = C_2M_2$

$5 \cdot \cancel{41.84}\ x = 41.84 \cdot 9.54$

$x = 41.84 \cdot 9.54 / 5$

$x = 79.83$

water to add = 79.83 - 41.84 = 37.99 g

15 wt% sample:

Current mass = 122.00 g

$x = 122.00 \cdot 9.54 / 15$

$= 77.59$

water to ~~add~~ evap = 122.00 - 77.59 = 44.41 g

25-1 sample:

Current mass = 136.53 g

$x = 136.53 \cdot 9.54 / 25$

$= 52.10$

water to evap = 136.53 - 52.10 = 84.43 g

25-2 sample:

Current mass = 136.32 g

$x = 13632 \cdot 9.54 / 25$

$x = 52.02$

water to evap = 136.32 - 52.02 = 84.30 g

calculations checked by S.A. Fitz 4/3/00 pumping process. When the target weight of the AZ-102 pretreated waste has been transferred, stop the pump, and reverse the peristaltic pumping direction to pump the composited AZ-102 pretreated waste that is in the tube back into the AZ-102 washed sludge jar. Finally, after all samples have been taken, clamp the end of the tube that was discharging into the stainless steel beaker or glass jar(s), remove the tubing from the peristaltic pump, and, after raising the clamped end of the tubing above the level of the jar, unclamp it allowing any remaining pretreated waste slurry to drain back into the jar.

9.49% ⊥ 9.58%

Weight of AZ-102 pretreated waste transferred from "AZ-102 Washed Solids Comp" to:

470.97 g "AZ-102 Washed Sludge Vit-1" stainless steel beaker

134.58 g "AZ-102 Washed Sludge: Rheol-5" 125 mL wide mouth glass jar

134.16 g "AZ-102 Washed Sludge: Rheol-15" 250 mL wide mouth glass jar

217.49 g "AZ-102 Washed Sludge: Rheol-25-1" 250 mL wide mouth glass jar

217.50 g "AZ-102 Washed Sludge: Rheol-25-2" 250 mL wide mouth glass jar

Sign and date Michael J. Schweiger 3/16/2000 Note: enter above data into Addendum 2, Table 1 below.

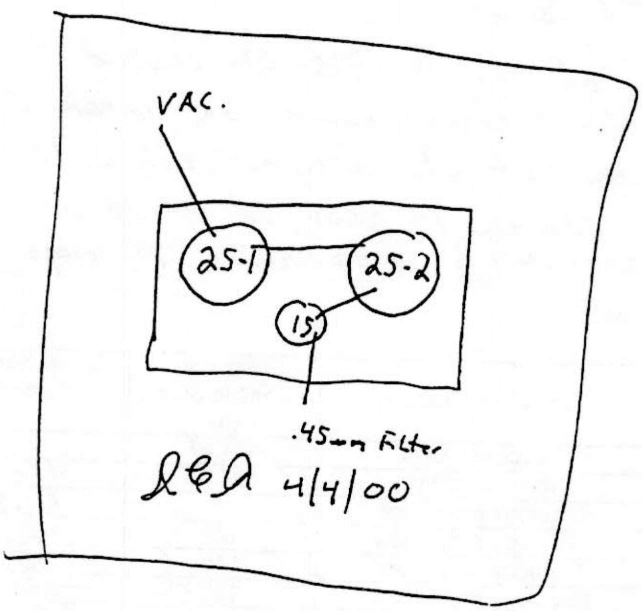
Observations: After sampling of the 2 AZ-102 Washed Solids Comp-ICP-1 (&2) plus the archieve sample on March 13, 2000, the AZ-102 Comp Waste was stored with a Teflon lid tightly capping it. On March 14, 2000, at ~ 10:00 am the container was weighed (2020.47g) to determine if evaporation OVER

Addendum 2, Table 1. Sample Designations

Sample ID	Sample Application	Nominal Sample Size (g)	Actual Sample Size (g)
AZ-102 Washed Solids Comp	Source material	954.57	
AZ-102 Washed Sludge Vit-1	Glass Batching	507.57	499.25
AZ-102 Washed Sludge: Rheol-5	Rheology properties	40	41.84
AZ-102 Washed Sludge: Rheol-15	Rheology properties	122	122.00
AZ-102 Washed Sludge: Rheol-25-1	Rheology properties	135	138.53 136-
AZ-102 Washed Sludge: Rheol-25-2	Rheology properties	135	~~135.84~~ 136-
AZ-102 Washed Solids Comp-ICP-1	wt% solids, ICP-AES	5	5.53
AZ-102 Washed Solids Comp-ICP-2	wt% solids, ICP-AES	5	6.21
AZ-102 Washed Sludge: Arch-1	Archiving	5	7.57

MJS 3/16/00

3'5



221.1
- 207.1
14.0 g/5hr ~ ~~0.8g/hr~~ ~3

11 g/

10) Measure the distance between the highest and lowest graduation on the ~~graduated glassware using a ruler.~~

High______ ml

Low______ ml

Distance______ cm

Cones used for settling study

used 10 ml glass graduated centrifuge cones cylindrical from 10 to ~3.5 ml
distance from 10-4ml = 4.2 cm

PRB 4/17/00

11) Transfer the required mass of material into the three graduated glassware.

	AZ EVAP A		AZ EVAP B		AZ EVAP C
Total	______g	Total	______g	Total	______g
Tare	______g	Tare	______g	Tare	______g
Slurry	______g	Slurry	______g	Slurry	______g

placed in vacuum train using current Bottles.

Samples 15, 25-1, & 25-2 placed on vacuum train Drilled hole in first cap and installed filter to keep out dirt.

12) Apply a vacuum to the samples. (ie same effect as blowing air over samples) PRB 4/5/00

13) Monitor the mass of each sample on a regular basis to assess the rate of evaporation. If necessary to accelerate evaporation, place the jar in an oven at 50°C (±10°C).

Rheol-15 ~~AZ EVAP A~~		-25-1 ~~AZ EVAP B~~		25-2 ~~AZ EVAP C~~		
Date	4/4/00	Date	4/4/00	Date	4/4/00	} starting mass
Mass	255.453	Mass	353.022	Mass	353222	
Date	4/5/00 7:50am	Date	4/5/00 8:00am	Date	4/5/00 8:05am	Restart 8:20am 4/5/00
Mass	244.720g	Mass	342.642	Mass	343.825	
Date	4/5/00 2:00pm	Date	4/6/00 8:05am	Date	4/6/00 8:05am	Restart 8:25 4/6
Mass	227.558g	Mass	315.706	Mass	316.880	
Date	4/6/00 7:45am	Date	4/7/00 8:30am	Date	4/7/00 8:30	
Mass	221.1128	Mass	295.061	Mass	295.037g	
Date	4/6/00 12:50pm	Date	4/7/00 3:40pm	Date	4/7/00 3:40pm	
Mass	207.164	Mass	282.987	Mass	277.592	
Date		Date	4/10/00 2:45	Date	4/10/00 2:45pm	
Mass		Mass	269.787	Mass	~~257.886~~ 257.886 PRB 4/10/00	
Date		Date		Date		
Mass		Mass		Mass		

25% samples look firm with cracked surfaces but look moist PRB 4/10/00

Battelle
Pacific Northwest Laboratories

ENGINEERING WORKSHEET

Prepared By: Paul Bredt | Date: 4/11/00 | Project: BNFL TP-29953-073

Subject: Water additions to AZ-102 samples Rheol-5, -15, -251, -25-2

	-5	-15	-25-1	-25-2
p6 step 9 Target mass (g)	214.41	211.75	269.59	269.52
Current mass (g)	175.987	206.909	269.582	257.778
water to add (g)	38.42	4.84	0.008	11.74
Water added (g)	38.638	5.087	0	11.725
final mass w/Jar (g)	214.625	~~211.96~~ 211.996 PRB 4/12/00	269.582	269.503

The above table documents water additions to AZ-102 samples following evaporation step 13 of Test plan

work performed on 4/12/00
PRB 4/12/00

Used Type 2 reagent water from Nanopure system in lab 201.
>17.6 M ohms

after final addition
Rheol-25-2

total 269.503 g
Tare 217.50 g
Slurry 52.003 g

~~AZ EVAP A~~ -15		~~AZ EVAP B~~ -25-1		~~AZ EVAP C~~ -25-2	
Date		Date		Date	
Mass		Mass		Mass	
Date		Date		Date	
Mass		Mass		Mass	

14) When the mass of each of the slurries reaches the targets calculated above, remove them from the vacuum and cap the sample. Record the mass. following water addition, see attached Bench Sheet PRB 4/12/00

~~AZ EVAP A~~ -05		~~AZ EVAP B~~ ~~-25-1~~ -15		~~AZ EVAP C~~ ~~-25-2~~ -25-1	
Total	214.625 g	Total	211.996 g	Total	269.582 g
Tare	134.58 g	Tare	134.16 g	Tare	217.49 g
Slurry	80.045 g	Slurry	77.836 g	Slurry	52.092 g

as per conversation with Stuart Arm on 4/10/00 PRB

15) If the samples are in glass jars, transfer ~5 ml (~8g) them to appropriately sized preweiged ~~graduated cylinders~~ 10 ml glass centrifuge cones with caps or plugs. Record the mass and volume of material in each of the graduated cylinders. centrifuge at ~1000xg for 5 min.

~~AZ A~~ Rheol-5 A		~~AZ B~~ Rheol-15 A		~~AZ C~~ Rheol-25-1 A	
Total	27.760 g	Total	29.582 g	Total	26.66 g
cone ~~Grad~~	19.968 g	cone ~~Grad~~	19.777 g	cone ~~Grad~~	19.848 g
Slurry	7.7920 g	Slurry	9.805 g	Slurry	6.81 g
Volume	~~7.67~~ 7.55 ml	Volume	~~7.2~~ 8.5 ml	Volume	5.4 ml

ρ=1.153

ρ = 1.26

~~ρ=1.025g/ml~~ (5 A) ρ= 1.03 g/ml PRB 4/20/00

sample Rheol-5 settling study conducted (steps 21-25) Before centrifugation. PRB 4/12/00

16) Preweigh 3 teflon coated magnetic stir bars.

A	B	C
g	g	g

17) Preweigh 3 appropriately sized volumetric flasks.

A	B	C
g	g	g

18) Place one stir bar in each of the volumetric flasks and fill to the mark with DI water. Record the mass of the volumetric flasks. Calculate the volume of the stir bars by the displacement of water.

A		B		C	
Total	g	Total	g	Total	g
Flask	( )g	Flask	( )g	Flask	( )g
Bar	( )g	Bar	( )g	Bar	( )g
Water	g	Water	g	Water	g

19) Place stir bar A in graduated cylinder **AZ A**, stir bar B in **AZ B**, and stir bar C in **AZ C**.

PR Bredt
12/6/99

AZ EVAP A		AZ EVAP B		AZ EVAP C	
Date	________	Date	________	Date	________
Mass	________	Mass	________	Mass	________
Date	________	Date	________	Date	________
Mass	________	Mass	________	Mass	________

14) When the mass of each of the slurries reaches the targets calculated above, remove them from the vacuum and cap the sample. Record the mass.

AZ EVAP A		AZ EVAP B		AZ EVAP C	
Total	________ g	Total	________ g	Total	________ g
Tare	________ g	Tare	________ g	Tare	________ g
Slurry	________ g	Slurry	________ g	Slurry	________ g

See step 15 page 8

15) If the samples are in glass jars, transfer them to appropriately sized preweiged ~~graduated cylinders~~ cones with B caps or plugs. Record the mass and volume of material in each of the graduated cylinders.

~~AZ A~~ Rheol 5B		~~AZ B~~ RHeol - 15B		~~AZ C~~ Rheol - 25B	
Total	27.658 g	Total	30.356 g	Total	26.58 g
cone ~~Grad~~	20.095 g	cone ~~Grad~~	19.836 g	cone ~~Grad~~	19.871 g
Slurry	7.563 g	Slurry	10.520 g	Slurry	6.71 g
Volume	7.2 ml	Volume	9.3 ml	Volume	5.5 ml
ρ	1.05 g/ml	ρ	1.131 g/ml	ρ	1.22 g/ml

16) Preweigh 3 teflon coated magnetic stir bars.

A	B	C
________ g	________ g	________ g

17) Preweigh 3 appropriately sized volumetric flasks.

A	B	C
________ g	________ g	________ g

18) Place one stir bar in each of the volumetric flasks and fill to the mark with DI water. Record the mass of the volumetric flasks. Calculate the volume of the stir bars by the displacement of water.

A		B		C	
Total	________ g	Total	________ g	Total	________ g
Flask	(________)g	Flask	(________)g	Flask	(________)g
Bar	(________)g	Bar	(________)g	Bar	(________)g
Water	________ g	Water	________ g	Water	________ g

19) Place stir bar A in graduated cylinder **AZ A**, stir bar B in **AZ B**, and stir bar C in **AZ C**.

20) Mobilize the material in each of the ~~graduated cylinders using a magnetic stir plate.~~ cones PRB 4/14/00 After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

Rheol-5A ~~AZ A~~ Time 2:42:30 Date 4/14/00

~~AZ B Time ______ Date ______~~

~~AZ C Time ______ Date ______~~

15 wt% & 25 wt% will not settle PRB 4/11/00

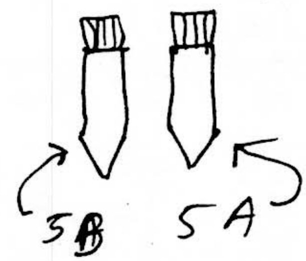
21) After turning off the stirrer, record the ambient temperature volume of settled solids layers and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day. Collect video during the first 3 days of settling.

Ambient 34 °C

Rheol-5A ~~AZ A~~ without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	4/14/00	2:47	7.75	7.75	0
15 min		2:57		7.75	
25 min		3:07		7.6	
35 min		3:17		7.5	
45 min		3:27		7.45	
55 min		3:37		7.45	
1 hour		3:42		7.4	
1.5 hour		4:12		7.3	
2 hour		4:42		7.2	
2.5 hour		5:12		7.1	
3 hour		5:42		7.0	
3.5 hour		6:12		6.9	
4 hour		6:42		6.8	
4.5 hour		7:12		6.7	
5 hour		7:42		6.55	
5.5 hour		8:12		6.5	
6 hour		8:42		6.4	
6.5 hour		9:12		6.3	
7 hour		9:42		6.2	
24 hour	4/15/00	2:42		4.75	
28 hour	4/15/00	6:42		4.7	
32 hour	4/15/00	10:42 pm		4.65	
48 hour	4/16/00	2:42 pm		4.55	
52 hour	4/16/00	6:42 pm		4.55	
56 hour	4/16/00	10:42 pm		4.55	
72 hr.	4/17/00	2:42		4.5	

one video, 5A is on the Right



Bottom of meniscus = 7.6 ml
top of meniscus = 7.75 ml

• Glass cones used for this study measure 4.2 cm from 10-4 ml

$\frac{4.2\ cm}{6\ ml}$ = ~~0.817 cm/ml~~ 0.700 cm/ml PRB 6/2/00 (Glass cones)

cones PRB 4/11/00

20) Mobilize the material in each of the ~~graduated cylinders using a magnetic stir plate~~. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

Rheol-5B ~~AZ A~~ Time ~~2:42~~ ~~2:42:30~~ 2:43:00 PRB 4/14/00 Date 4/14/00

~~AZ B Time ______ Date ______~~

~~AZ C Time ______ Date ______~~

15 & 25 wt% will not settle

21) After turning off the stirrer, record the ambient temperature volume of settled solids layers and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day. Collect video during the first 3 days of settling.

Ambient 34 °C

Rheol-5B
~~AZ A~~ without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	4/14/00	2:47	7.65	7.65	
15 min	↓	2:57	↓	7.4	
25 min		3:07		7.25	
35 min		3:17		7.15	
45 min		3:27		7.05	
55 min		3:37		7.0	
1 hour		3:42		6.9	
1.5 hour		~~4~~3:12		6.6	
2 hour		4:42		6.4	
2.5 hour		5:12		6.2	
3 hour		5:42		~~7~~6.05	
3.5 hour		6:12		~~5~~6.9	
4 hour		6:42		~~5~~6.8	
4.5 hour		7:12		5.65	
5 hour		7:42		5.55	
5.5 hour		8:12		5.45	
6 hour		8:42		5.35	
6.5 hour		9:12		5.3	
7 hour	↓	9:42		5.2	
24 hour	4/15/00	2:4~~5~~2		4.4	
28 hour	↓	6:4~~5~~2		4.4	
32 hour	↓	10:4~~5~~2		4.35	
48 hour	4/16/00	2:4~~5~~2		4.3	
52 hour	↓	6:42		4.3	
56 hour	↓	10:42	↓	4.3	

72 hrs 4/17/00 2:42 4.3

4/20/00
PRB
7.2

Bottom of meniscus = ~~7.4~~ ~~7.3~~ ml

top of meniscus = 7.65 ml

(glass cones)

AZ B without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

will not settle
PRB 4/4/00

AZ C without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

will not settle
4/4/00

22) Place the ~~graduated cylinders~~ centrifuge cones in an oven at 50°C (±5°C)for 1 day. Remove the samples and record the mass and volume.

4/27/00
after
3 days @ 50°C
PRB

	Rheol-5 A ~~AZ A~~	-15 A ~~AZ B~~	-25 ~~A~~ ~~AZ C~~ B
Total	27.712 g	29.018 g	26.326 g
Tare	19.968 g	19.777 g	19.871 g
Slurry	7.744 g	9.241 g	6.455 g
Volume	7.6 ml	8.3 ml	5.4 ml
	ρ=1.02	ρ=1.11	1.20

Some Bubbles released from 5A/B during level measurement PRB 4/27/00

	-5 B	-15 B	-25 A
Total	27.189 g	30.171 g	26.367 g
Tare	20.095 g	19.836 g	19.848 ~~19.871~~ PRB 4/27/00 g
slurry	7.094 g	10.335 g	6.519 g
volume	6.8 ml	9.2 ml	5.3 ml
	ρ:1.04	ρ-1.12	ρ:1.23

Page 12A PRB 4/11/00

23) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

RLD-1-5A ~~AZ A~~ Time 9:00am Date 5/2/00

~~AZ B Time Date~~

~~AZ C Time Date~~

15 + 25 wt% will not settle PRB 4/11/00

24) Return the graduated cylinders to the oven at 50°C.

25) After turning off the stirrer, record the volume of settled solids layers and liquids every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

RLD-1-5A
~~AZ A~~ without glass formers at 50°C

	Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
1/2	30 min	5/2/00	9:30	7.8	7.5	0.3
1	60 min	↓	10:00	7.8	7.4	0.4
1.5	90 min		10:30	7.8	7.1	0.7
2	120 min		11:00	7.8	7.0	0.8
2.5	150 min		11:30	7.8	6.8	1.0
3	180 min		12:00	7.8	6.6	1.2
3.5	210 ~~hour~~ min		12:30	7.8	6.5	1.3
4	240 ~~hour~~ min		1:00	7.8	6.3	1.5
4.5	270 ~~hour~~ min		1:30	7.8	6.2	1.6
5	300 ~~hour~~ min		2:30	7.8	5.9	1.9
	24 hour	5/3/00	9:30	7.8	4.8	3.0
	28 hour	↓	11:50	7.8	4.7	3.1
	32 hour		3:15	7.8	4.7	3.1
	48 hour	5/4/00	8:00	7.8	4.7	3.1
	52 hour	↓	1:00	7.8	4.7	3.1
	56 hour			7.8	4.7	

total - 7.8 ml top of meniscus

7.6 bottom of meniscus

23) Mobilize the material in each of the ~~graduated cylinders using a magnetic stir plate~~ 5 wt% concs. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

~~AZ A~~ Rheol.-5B Time 9:00 am Date 5/2/00

~~AZ B Time _____ Date _____~~

~~AZ C Time _____ Date _____~~

15 & 25 wt% will Not settle PRB 4/11/00

24) Return the graduated cylinders to the oven at 50°C.

25) After turning off the stirrer, record the volume of settled solids layers and liquids every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

Rheol 5B
~~AZ A~~ without glass formers at 50°C

Target	Date	time	Total (ml) PRB	Solid (ml)	Liquid (ml)
30 min	5/2/00	9:30	~~6.9~~ 7.2	6.6	
60 min	↓	10:00	↓	6.5	
90 min		10:30		6.3	
120 min		11:00		6.2	
150 min		11:30		6.05	
180 min		12:00		5.95	
210 hour		12:30		5.8	
240 hour		1:00		5.7	
270 hour		1:30		5.6	
300 hour	↓	2:~~00~~ 30 PRB		5.3	
24 hour	5/3/00	9:30		4.7	
28 hour	↓	11:56		4.6	
32 hour		3:15		4.6	
48 hour	5/4/00	8:00 am		4.5	
52 hour	↓	1:00 pm		4.5	
56 hour	↓		↓	4.5	

top of meniscus: ~~6.9~~ 7.2 ml
Bottom of meniscus: 6.7 ml

AZ B without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

AZ C without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

will not
settle
PRB 4/4/00

26) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to 1000 s^{-1}. Print out a copy of the rheogram and attach to this test plan.

Viscometer M5 Location A-Cell south Geometry MVI "PNL000 2927"

Viscosity 95.5 cP Lot 111199 Manufacturer controller exp. 3/01 Brookfield

File name 050200 A B C Date analyzed 5/2/00

27) Remove the graduated cylinders from the oven and allow to cool overnight.

28) While stirring the samples on a magnetic stir plate remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to 300 s^{-1} over 2 minutes and then returning from 300 to 0s^{-1} over 2 minutes. Print out a copy of the rheograms and attach to this test plan.

Confirmed on 4/7/00 with S.A. That 300 s-1 is appropriate max shear rate. PRB 4/11/00

	50°C	25°C	
-5 ~~AZ A~~	050800H	File name 050800A	Date analyzed
~~AZ A~~ Duplicate	050800I	File name 050800B 05800C	Date analyzed
~~AZ B~~	050800J	File name 050800D	Date analyzed
-15 ~~AZ B~~ Duplicate	050800K	File name 050800E	Date analyzed
~~AZ C~~	050800L	File name 050800F	Date analyzed
-25 ~~AZ C~~ Duplicate	050800M	File name 050800G	Date analyzed

C-104 samples, not AZ-10- should be listed under step 91

~~29) Return this material to the respective graduated cylinders.~~

29) Rinse this material Back into sample jars.

30) Also Rinse contents of centrifuge cones Back into respective jars.

31) Re weigh the sample Jars.

	Rheol-5	Rheol-15	Rheol-25-1
mass	____ g	____ g	____ g

32) evaporate samples back to target weights listed under step on page 6. Record new masses

	Rheol-5	Rheol-15	Rheol-25-1
Mass	____ g	____ g	____ g

(over)

See attachment following step 92 - PRB 5/31/00 p14 (Back)

On 6/15/00 the AZ-102-?S-1 was diluted to ~~25~~ 20wt%, total mass = ~~282.6~~ g PRB 6/15/00
282.7g

- Sample was removed for Rheology, New total mass
~~20~~
270.1g

- Manipulator Broke recovering sample from cup and most of material rinsed from Rheometer was lost on deck.

total loss = 282.7 - 270.1g
= 12.6g of diluted

~~12.6g · 2.5~~
PRB 6/5/00
~~0.2 wt%~~

$12.6g \cdot 0.2 \frac{solid}{slurry} = 2.5g$ solids

- During Rheology on 25°C 20wt% solids
  - Material stuck to sides of Jar so unable to get a good subsample
  -

Run	File
Sample 1	060500A
Sample 1 dup	060500B
Sample 2	060500C

0 - 300 sec^{-1} over 2 min
300 - 0 sec^{-1} over 2 min
25°C

Paul B[illegible] 6/15/00

Test Plan 29953-073
Back of page 14

Hand Calculation Check

Glass formulations in this report for AW-101 and AN-107 are considered to be proprietary by VSL/Duratek

	A	B	C	D	E	F	G	H	I
1	water adjust 052300.xls								
2	36669								
3	Paul Bredt		Slurry wt% solids						
4			9.535						
5									
6					Rheol-25-1		Rheol-25-2		
7	Sample	Slurry Basis	Rheol-5	Rheol-15	For 25 wt%	For 20 wt%	For 25 wt%	For 20 wt%	For 15 wt%
8	Slurry Mass (g)	436.69	41.84	122	136.53	136.53	136.32	136.32	136.32
9	Sr/TRU Precipitate (25 wt% solids basis)	19.21	1.841	5.367	6.006	6.006	5.997	5.997	5.997
10	Actual Cs Eluant (Diluted Basis)	137.83	13.206	38.506	43.092	43.092	43.026	43.026	43.026
11	Actual Tc Eluant (Diluted Basis)	76.67	7.346	21.42	23.971	23.971	23.934	23.934	23.934
12	Na2B4O7•10H2O	10.76	1.031	3.006	3.364	3.364	3.359	3.359	3.359
13	LiOH•H2O	15.51	1.486	4.333	4.849	4.849	4.842	4.842	4.842
14	Na2SiO3•5H2O	32.09	3.075	8.965	10.033	10.033	10.017	10.017	10.017
15	SiO2 (Sil-co-Sil 75)	39.73	3.807	11.1	12.421	12.421	12.402	12.402	12.402
16	Sugar (Granular)	1.34	0.128	0.374	0.419	0.419	0.418	0.418	0.418
17									
18									
19	Initial solids		=C8*0.0954	=D8*0.0954	=E8*0.0954	=F8*0.0954	=G8*0.0954	=H8*0.0954	=I8*0.0954
20	Initial Water		=C8-C19	=D8-D19	=E8-E19	=F8-F19	=G8-G19	=H8-H19	=I8-I19
21	Total water needed		=(1-0.05)*C19/0.05	=(1-0.15)*D19/0.15	=(1-0.25)*E19/0.25	=(1-0.2)*F19/0.2	=(1-0.25)*G19/0.25	=(1-0.2)*H19/0.2	=(1-0.15)*I19/0.15
22	Water to add		=C21-C20	=D21-D20	=E21-E20	=F21-F20	=G21-G20	=H21-H20	=I21-I20
23	Adjusted mass		=C8+C22	=D8+D22	=E8+E22	=F8+F22	=G8+G22	=H8+H22	=I8+I22
24	Bottle Tare		134.58	134.16	217.49	217.49	217.5	217.5	217.5
25	Total Target		=C24+C23	=D24+D23	=E24+E23	=F24+F23	=G24+G23	=H24+H23	=I24+I23

- Equations match across columns
- Checked values for column E and found them to agree with spreadsheet values.
- Verified equation for row 21.

Harry Smith
5/23/00

These calculations ~~were performed~~ to determine the water required to reach the client specified wt% solids values.
Paul Bredt
5/25/00

Plan 29953-073
PRB 5/31/00

Glass formulations in this report for AW-101 and AN-107 are considered to be proprietary by VSL/Duratek

Page of 2
Harry D. Smith
5/23/00

	A	B	C	D	E	F	G	H	I
1	water adjust 052300.xls								
2	5/23/2000								
3	Paul Bredt		Slurry wt% solids						
4			9.535						
5									
6					Rheol-25-1		Rheol-25-2		
7	Sample	Slurry Basis	Rheol-5	Rheol-15	For 25 wt%	For 20 wt%	For 25 wt%	For 20 wt%	For 15 wt%
8	Slurry Mass (g)	436.69	41.84	122	136.53	136.53	136.32	136.32	136.32
9	Sr/TRU Precipitate (25 wt% solids	19.21	1.841	5.367	6.006	6.006	5.997	5.997	5.997
10	Actual Cs Eluant (Diluted Basis)	137.83	13.206	38.506	43.092	43.092	43.026	43.026	43.026
11	Actual Tc Eluant (Diluted Basis)	76.67	7.346	21.42	23.971	23.971	23.934	23.934	23.934
12	Na2B4O7•10H2O	10.76	1.031	3.006	3.364	3.364	3.359	3.359	3.359
13	LiOH•H2O	15.51	1.486	4.333	4.849	4.849	4.842	4.842	4.842
14	Na2SiO3•5H2O	32.09	3.075	8.965	10.033	10.033	10.017	10.017	10.017
15	SiO2 (Sil-co-Sil 75)	39.73	3.807	11.1	12.421	12.421	12.402	12.402	12.402
16	Sugar (Granular)	1.34	0.128	0.374	0.419	0.419	0.418	0.418	0.418
17									
18									
19	Initial solids		3.992	11.639	13.025	13.025	13.005	13.005	13.005
20	Initial Water		37.848	110.361	123.505	123.505	123.315	123.315	123.315
21	Total water needed		75.839	65.953	39.075	52.100	39.015	52.020	73.695
22	Water to add		37.991	-44.408	-84.430	-71.405	-84.300	-71.295	-49.620
23	Adjusted mass		79.831	77.592	52.100	65.125	52.020	65.025	86.700
24	Bottle Tare		134.580	134.160	217.490	217.490	217.500	217.500	217.500
25	Total Target		214.411	211.752	269.590	282.615	269.520	282.525	304.200

Attachment to test plan 29953-073
PRB 5/31/00

Glass formulations in this report for AW-101 and AN-107 are considered to be proprietary by VSL/Duratek

Attachment to Test Plan BNFL-29953-073

calculations I performed following loss of some material from sample Rheol-25-1 as described on the back of page 14 of the test instruction.

Paul Bredt 6/7/00

water adjust 052300.xls
6/7/00
Paul Bredt

Slurry wt% solids
9.535

				Rheol-25-1		Rheol-25-1 after spill		Rheol-25-2		
Sample	Slurry Basis	Rheol-5	Rheol-15	For 25 wt%	For 20 wt%	for 20wt%	For 15 wt%	For 25 wt%	For 20 wt%	For 15 wt%
Slurry Mass (g)	436.69	41.84	122	136.53	136.53	110.31	110.31	136.32	136.32	136.32
Sr/TRU Precipitate (25 wt% solids basis)	19.21	1.841	5.367	6.006	6.006	4.853	4.853	5.997	5.997	5.997
Actual Cs Eluant (Diluted Basis)	137.83	13.206	38.506	43.092	43.092	34.817	34.817	43.026	43.026	43.026
Actual Tc Eluant (Diluted Basis)	76.67	7.346	21.42	23.971	23.971	19.367	19.367	23.934	23.934	23.934
Na2B4O710H2O	10.76	1.031	3.006	3.364	3.364	2.718	2.718	3.359	3.359	3.359
LiOHH2O	15.51	1.486	4.333	4.849	4.849	3.918	3.918	4.842	4.842	4.842
Na2SiO35H2O	32.09	3.075	8.965	10.033	10.033	8.106	8.106	10.017	10.017	10.017
SiO2 (Sil-co-Sil 75)	39.73	3.807	11.1	12.421	12.421	10.036	10.036	12.402	12.402	12.402
Sugar (Granular)	1.34	0.128	0.374	0.419	0.419	0.338	0.338	0.418	0.418	0.418
Initial solids		3.992	11.639	13.025	13.025	10.524	10.524	13.005	13.005	13.005
Initial Water		37.848	110.361	123.505	123.505	99.787	99.787	123.315	123.315	123.315
Total water needed		75.839	65.953	39.075	52.100	42.095	59.634	39.015	52.020	73.695
Water to add		37.991	-44.408	-84.430	-71.405	-57.693	-40.153	-84.300	-71.295	-49.620
Adjusted mass		79.831	77.592	52.100	65.125	52.618	70.158	52.020	65.025	86.700
Bottle Tare		134.580	134.160	217.490	217.490	217.490	217.490	217.500	217.500	217.500
Total Target		214.411	211.752	269.590	282.615	270.108	287.648	269.520	282.525	304.200

sludge only (no glass or 2ndary waste)

270.153

282.515 g

46.133 g Tc eluant

25.116 PRB 6/9/00

86.529 g Cs eluant

Attachment to test
Plan 29953-073
PRB 5/31/00

Paul Bredt
5/25/00

AN-107 Sr/TRU for AZ-102

The AN-107 Sr/TRU was tested for wt% solids under Test Instruction BNFL-TI-29953-079-1 Rev 1 addendum 6. The wt% solids were determined to be 46.26%. These solids ~~were in~~ are in Jar "AN-107 DF Solids". This jar tare is 217.309 g. During wt% solids work, the jar weighed 227.84 g

227.84 g - 217.309 g = 10.53 g

The jar weighed on 5/25/00 226.64 g

226.64 g - 217.309 g = 9.33 g

So... Values for Sr/TRU addition to AZ-102 need to be corrected by $\frac{9.33}{10.53} = 0.886$

In ~~appendix~~ addendum to TP 29953-079, the mass to add to AZ-102 Feed was 10.38 g. This should now be 10.38 g · 0.886 = 9.20 g

New solids content is 52.20 wt%

The Following table is to be used for the addition of Sr/TRU to AZ-102 samples in grams. Sr/TRU solids will be added and supplimented with water.

Slurry Basis		Rheol-5	Rheol 15	Rheol 25-1	Rheol 25-2
slurry mass	436.69	41.84	122.00	136.53	136.32
Sr/TRU slurry	~~9.92~~ 9.20 ~~x 46.26/25 = 17.02~~	~~1.63~~	~~4.75~~	~~5.32~~	~~5.31~~
	PRB 9.20 x 52.20/25 = 19.21	1.84	5.37	6.01	6.00
Sr/TRU solids	5/25/00 9.20	0.88	2.57	~~2.88~~ 2.37	2.87
water	~~7.92~~	~~0.75~~	~~2.18~~	~~2.44~~	~~2.44~~
	10.01	0.96	2.80	3.13	3.13

PRB 5/25/00

After spill PRB 6/9/00

Calculations & Method reviewed by Harry D. Smith 5/25/00

30) Add the BNFL prescribed quantity of intermediate waste products to each of the three graduated cylinders and stir for 1 hour. Use the space below to record the quantity of intermediate waste products added and the time and date stirring started and stopped. 5/25/00 [illegible]

Rheol-5
~~AZ-A~~ Stir Start (time/date) 10:15 Stir Stop (time/date) 10:48

Source Chemical	Target wt%	Amount (g)	Actual Added (g)
Actual Sr/Tru		0.88 (g)	0.872
Glass former mix		9.527 g	9.454
H_2O for Sr/Tru		0.96	See below
H_2O total		2.01 g	2.2 g

old wt. 214.46 g
Start wt. 214.290 g
Aft. Sr/Tru 215.162 g
Aft. G.F 224.616 g
water added - 0.19 + 0.96 + ? 0.
weight aft. stirring 223.736 g
wt. aft H_2O 225.936 g

* Cell evaporation wt may be a small factor of error in actual wt additions

Rheol-15
~~AZ-B~~ Stir Start (time/date) 11:20 Stir Stop (time/date) 11:55

Source Chemical	Target wt%	Amount (g)	Actual Added (g)
Actual Sr/Tru		2.57 (2.61)	2.495
Glass Former mix		27.778	27.428
H_2O for Sr/Tru		2.80	See below
H_2O total		5.053	5.06

old wt 211.73 g
start wt 211.340 g
Sr/Tru add 213.835 g
GF mix add 241.263 g
wt. aft. stir 239.403 g
H_2O add 0.39 + 2.80 + 1.86
aft. 244.460 g

Rhel-25-1
~~AZ C~~ Stir Start (time/date)________ Stir Stop (time/date)________

Source Chemical	Target wt%	Amount (g)	Actual Added (g)

Not Needed, see attached email from Stuart Arm PRB 5/31/00

31) Cap the graduated cylinders and record the mass and volume.

AZ A		AZ B		AZ C	
Total	________g	Total	________g	Total	________g
Tare	________g	Tare	________g	Tare	________g
Slurry	________g	Slurry	________g	Slurry	________g
Volume	________ml	Volume	________ml	Volume	________ml

32) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

AZ A Time ________ Date________

AZ B Time ________ Date________

AZ C Time ________ Date________

~~Graduated~~ centrifuge cones used. ~~See~~ step 94 PRB 5/31/00

Not Needed PRB 5/31/00

Attachment to test plan 29953-073 PRB 5/31/00

p1 of 2

Bredt, Paul R

From: Stuart Arm [sarm@bnflinc.com]
Sent: Tuesday, May 23, 2000 9:56 AM
To: Bredt, Paul R
Cc: Smith, Gary; Morrey, Eugene V; Kurath, Dean E; Jagoda, Lynette K
Subject: Re: Sr/TRU Simulant

Paul,

Yes, please proceed as you describe.

Thanks,
Stuart.

"Bredt, Paul R" wrote:

Stuart,

We had planned to repeat the AZ-102 25wt% rheology at 50°C. We could omit this data point (25°C results are coming out similar to 50°C) and instead run the sample diluted at 20wt%.

If this sounds acceptable, please confirm this summary of modifications:

1) Omit the AZ-102 25wt% rheogram at 50°C
2) Dilute the AZ-102 25wt% sample to 20wt% and analyze for shear stress versus shear rate at 25°C
3) Omit AZ-102 25wt% sample from glass former addition testing.
4) Use AZ-102 15 wt% sample for mixing and aging study
5) Leave C-104 testing as planned with 25wt% sample for mixing and aging study.

Thanks,
Paul

-----Original Message-----
From: Stuart Arm [SMTP:sarm@bnflinc.com]
Sent: Tuesday, May 23, 2000 9:41 AM
To: Bredt, Paul R
Cc: Smith, Gary; Morrey, Eugene V; Kurath, Dean E
Subject: Re: Sr/TRU Simulant

Paul,

Please conduct the mixing and aging study with the 15 wt% slurry and dilute the
25
wt% slurry to 20 wt% and determine the yield stress and viscosity (if no cost impact).

Thanks,
Stuart.

"Bredt, Paul R" wrote:

Stuart,

We have finished the AZ-102 and C-104 melter feed testing (no glass formers or

Attachment to Test Plan 29953-073 PRD 5/31/00 p 2 of 2

secondary waste products). The table below summarizes some of the rheology results at 25°C. Data at 50°C is virtually identical. We are becoming a bit concerned about adding glass formers to the AZ-102 25wt% sample. This may become too thick to handle (it is almost already too thick to handle). Do you want us to continue as planned with mixing and aging studies on the 25wt% AZ-102, or switch to the 15wt% sample? My feeling is we may want to eliminate the AZ-102 25wt% sample from the test matrix altogether or dilute it down significantly given this rheology data.

Thanks,
Paul

Sample	Yield Stress (Pa)	Viscosity 100 s-1 (cP)
C-104 5wt%	<0.5	<5
C-104 15wt%	0.5	6
C-104 25 wt%	6	63
AZ-102 5wt%	0.5	7
AZ-102 15wt%	15	200
AZ-102 25 wt%	190	2000

-----Original Message-----
From: Stuart Arm [SMTP:sarm@bnflinc.com]
Sent: Tuesday, March 14, 2000 3:14 PM
To: Bredt, Paul R
Subject: Re: Sr/TRU Simulant

Paul,

Use 25 wt% solids concentration (this should be the same as the waste).

Stuart.

"Bredt, Paul R" wrote:

> Stuart,
>
> As we discussed a few weeks ago, we have made a Sr/TRU precipitate simulant from
> the AN-107 simulant. We are having this analyzed by ICP and should have results
> in a week or so. Based on the VSL formulation, we plan to add 25.674 grams of
> this Sr/TRU precipitate on a dry basis for every 400 g C-104 waste (20wt%)
> during the rheological testing.
>
> Question: What wt% water should we shoot for in the Sr/TRU precipitate? (i.e.
> how much water should we use to suspend our 25.674 grams of precipitate)
>
> Thanks,
> Paul

33) After turning off the stirrer, record the ambient temperature, volume of settled solids layers and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day. Collect video during the first 3 days of settling.

Ambient____________°C

AZ A with waste products at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See step ~~44~~ 31
PRB
5/31/00

~~**AZ B** with waste products at ambient temperature~~

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See step 31
PRB
5/31/00

AZ C with waste products at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

34) Place the graduated cylinders in an oven at 50°C (±5°C) for 1 day. Remove the samples and record the mass and volume.

	AZ A		**AZ B**		**AZ C**
Total	________g	Total	________g	Total	________g
Tare	________g	Tare	________g	Tare	________g
Slurry	________g	Slurry	________g	Slurry	________g
Volume	________ml	Volume	________ml	Volume	________ml

35) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

AZ A Time __________ Date __________

AZ B Time __________ Date __________

AZ C Time __________ Date __________

See step 31
PRB
5/31/00

36) Return the graduated cylinders to the oven at 50°C.

37) After turning off the stirrer, record the volume of settled solids layers and liquids every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

AZ A with intermediate waste products at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

AZ B with intermediate waste products at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

Not Needed
see step 31
PRB
5/31/00

AZ C with intermediate waste products at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

38) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to approximately 1000 s^{-1}. Print out a copy of the rheogram and attach to this test plan.

Viscometer____________ Location_____________ Geometry

Viscosity___________cP Lot_______________ Manufacturer

File name ___________ Date analyzed

39) Remove the graduated cylinders from the oven and allow to cool overnight.

40) While stirring the samples on a magnetic stir plate remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to 300 s^{-1} in 2 minutes and then returning from 300 to 0s^{-1} in 2 minutes. Print out a copy of the rheograms and attach to this test plan.

AZ A File name _________ Date analyzed

AZ A Duplicate File name _________ Date analyzed

AZ B File name _________ Date analyzed

AZ B Duplicate File name _________ Date analyzed

AZ C File name _________ Date analyzed

See step 31
PRB
5/31/00

AZ C Duplicate File name __________ Date analyzed

41) Return this material to the respective graduated cylinders.

42) Add the BNFL prescribed quantity of glass formers to each of the three graduated cylinders and stir for 1 hour. Use the space below to record the quantity of glass formers added and the time and date stirring started and stopped.

AZ A Stir Start (time/date)_________ Stir Stop (time/date)_________

Glass Formers:

Source Chemical	Manufacturer	Lot#	Target wt%	Amount (g)	Actual Added (g)

See step 30
PRB
5/31/00

AZ B Stir Start (time/date)_________ Stir Stop (time/date)_________

Glass Formers:

Source Chemical	Manufacturer	Lot#	Target wt%	Amount (g)	Actual Added (g)

AZ C Stir Start (time/date)_________ Stir Stop (time/date)_________

Glass Formers:

Source Chemical	Manufacturer	Lot#	Target wt%	Amount (g)	Actual Added (g)

See step 30
PRB 5/31/00

43) Cap the graduated cylinders and record the mass and volume.

AZ A		AZ B		AZ C	
Total	____________g	Total	____________g	Total	____________g
Tare	____________g	Tare	____________g	Tare	____________g
Slurry	____________g	Slurry	____________g	Slurry	____________g
Volume	____________ml	Volume	____________ml	Volume	____________ml

44) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

AZ A Time ____________ Date ____________

AZ B Time ____________ Date ____________

AZ C Time ____________ Date ____________

run in graduated cones. See step with C104 Batch. 94.
PRB 5/31/00

45) After turning off the stirrer, record the ambient temperature, volume of settled solids layers and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day. Collect video during the first 3 days of settling.

Ambient____________°C

AZ A with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See step 94
PRB
5/31/00

AZ B with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See step 94
PRB 5/31/00

AZ C with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See step 94
PRB 5/31/00

46) Place the graduated cylinders in an oven at 50°C for 1 day. Remove the samples and record the mass and volume.

AZ A

Total ____________g
Tare ____________g
Slurry ____________g
Volume ____________ml

AZ B

Total ____________g
Tare ____________g
Slurry ____________g
Volume ____________ml

AZ C

Total ____________g
Tare ____________g
Slurry ____________g
Volume ____________ml

See step 94
Samples analyzed with a batch of C104 samples and data is recorded together PRB 8/10/00

47) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

AZ A Time ____________ Date______________

AZ B Time ____________ Date______________

AZ C Time ____________ Date______________

48) Return the graduated cylinders to the oven at 50°C.

49) After turning off the stirrer, record the volume of settled solids layers and liquids every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

AZ A with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See step 46

AZ B with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

AZ C with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See step 46

50) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to approximately 1000 s^{-1}. Print out a copy of the rheogram and attach to this test plan.

Viscometer Haake M5 Location A-Cell Geometry 8 MVT

Viscosity 95.5 cP Lot 111199 Manufacturer Brookfield

File name 060100A
B
C

Date analyzed 6/1/00

51) Remove the graduated cylinders from the oven and allow to cool overnight.

52) While stirring the samples on a magnetic stir plate remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to 300 s^{-1} in 2 minutes and then returning from 300 to 0s^{-1} in 2 minutes. Print out a copy of the rheograms and attach to this test plan.

Files 25°C Date

AZ A	File name ______	Date analyzed	5wt% 060200A	6/2/00
AZ A Duplicate	File name ______	~~Date analyzed~~	B	
			C	
AZ B	File name ______	Date analyzed	15wt% 060200D	
			E	
AZ B Duplicate	File name ______	Date analyzed		
			50°C	
AZ C	File name ______	Date analyzed	5wt% 060700A	6/7/00
			B	
AZ C Duplicate	File name ______	Date analyzed	15wt% 060700C	
			D	
			E	

53) Return this material to the respective graduated cylinders.

54) Assemble a mixing vessel using the following parts or equivalent. Attach impeller to a stirring motor capable of maintaining a constant rotational rate from 100-1400 rpm.

Part	Vendor	Catalog number
500 ml O-ring Sealed Kettle, 3.75 inch OD, 5 3/8 inch flange	Labglass	LG-8071-100
Clamp	Labglass	LG-7316-106
O-ring	Labglass	LG-1022-476
Kettle top with three 24/40 necks	Labglass	LG-8072-100
2 3/8 diameter 4 blade impeller	Fisher Scientific	14-505-20G

used 2 blade folding impell.

Tare 168.05 g Labeled "1"
Can to hold round bottom 67.76 g Labeled mix
Stopper Labeled "1" 15.45 g
"2" 15.42 g

Target mass for AZ-102 ... in round bottom
86.7g of slurry
168.1g for vessel
67.8g for ...
322.6g target total

3-6481

. evaporated in air, mass starting mixing study = 322.580g on 6/19/00

55) Transfer the sample specified by BNFL to the mixing vessel (do not transfer the stir bar). Record which sample was transferred as well as the day and time transferred below.

Sample transferred AZ-102 Rheol-25-2 Date 6/13/00 Time 9:30am

56) Turn on the stirrer and adjust the rotational speed to that specified by BNFL. Record the time, date and speed below. used NON-PNNL NIST traceable Tachometer Fisher Scientific model 05-028-24 serial L710985 exp 4-26-01

Speed 327 rpm Date 6/19/00 Time 2:10pm

To check cal, I held tach to fluorescent lamp and read 7200 RPM

added 31.071g of AZ-102 ... former mix

57) After 1 hour of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to 300 s^{-1}. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name 061900A Date analyzed 6/19/00 3:10pm

Run 2 File name 061900B Date analyzed 6/19/00

58) After 1 day of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to 300 s^{-1}. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name 062000A Date analyzed 6/20/00

Run 2 File name 062000B Date analyzed 6/20/00

59) After 1 week of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to 300 s^{-1}. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name 062600A Date analyzed 6/26/00

Run 2 File name 062600B Date analyzed 6/26/00

sample transferred and evaporated to target by a flow of Air

60) After removing the last sample (after 1 week of stirring), transfer the sample back to the graduated ~~cylinder~~ Jar. Record sample ID, volume and mass of material.

AZ-102 7/20/00

Total	g	Total	100	ml
Tare	217.61 g	Solids	80	ml
Slurry	g	Liquids	20	ml

Target slurry mass 86.7g
former 31.1g
Jar 217.61g
335.41g ← target mass

61) Focus a video camera on the solids-liquid interface of the sample and collect video for one week taking care not to disturb the sample. Report any observed gas retention and/or release behavior to the cognizant scientist. Camera not available

62) After the sample has remained undisturbed for 1 week, remove the standing liquid using a glass

- Sample in jar agitated on 7/20/00 @ 2:02pm
- on 7/25/00 solids at 92 ml mark, recorded interface for 2 hr from 9:30-11:30am
- on 7/26/00 solids at 92 ml mark, no retained Bubbles observed
- 1 week - on 7/27/00 solids @ ~91-92 mark - no bubbles ... (... pm)

pipette:

63) Gently collect a subsample of the settled solids and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to 300 s^{-1}. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name 072800A Date analyzed 7/28/00

Run 2 File name 072800B Date analyzed 7/28/00

072800C

C-104 samples received from Vin [illegible] on 4/24/00.
samples are "C-104 washed Solids Rheol-5"
-15
-25-1
-25-2

Tare weights are not available, but TP BNFL-29953-79 Rev. 1 addendum 1 list masses as follows
-5, 17.4 g   -25-1, 85.0 g
-15, 51.8 g   -25-2, 85.2 g

C-104

64) Record the solids concentration of the C-104 feed.

Wt% solids= 20 (C1)   Data Source TP-29953-79 Rev. 1

65) Record the Wt% solids targets provided by BNFL.

Wt% solids = 5 (C2a)

Wt% solids = 15 (C2b)

Wt% solids = 25 (C2c)

Due to lack of tare wts, samples Transferred to New Jars

Jar	Tare (g)
C104 5	133.1223
C104 15	134.2137
C104 25-1	134.2422
C104 25-2	133.9558

PRB 4/24/00

DI water used to wash solids into New Jars

66) Load 8 ml of C-104 material into 5 preweighed centrifuge cones. Record the new weight.

C6 1
Total ____________g
Cone ____________g
Slurry ____________g

C6 2
Total ____________g
Cone ____________g
Slurry ____________g

C6 3
Total ____________g
Cone ____________g
Slurry ____________g

C6 4
Total ____________g
Cone ____________g
Slurry ____________g

C6 5
Total ____________g
Cone ____________g
Slurry ____________g

67) Remove the caps, and place the centrifuge cones in a vacuum jar. If necessary to accelerate evaporation, place the jar in an oven at 50°C (±10°C).

68) At intervals determined by the cognizant scientist, remove the cones from the vacuum jar, replace the caps, and record the new weight.

C6 1
Total ____________g
Cone ____________g
Slurry ____________g

C6 2
Total ____________g
Cone ____________g
Slurry ____________g

C6 3
Total ____________g
Cone ____________g
Slurry ____________g

C6 4
Total ____________g
Cone ____________g
Slurry ____________g

C6 5
Total ____________g
Cone ____________g
Slurry ____________g

See step 3
PRB 4/24/00

69) Centrifuge the cones at ~1000 x g for one hour. Record the solids and liquid volumes.

C6 1

Total ____________ ml
Solid ____________ ml
Liquid ____________ ml

C6 2

Total ____________ ml
Solid ____________ ml
Liquid ____________ ml

C6 3

Total ____________ ml
Solid ____________ ml
Liquid ____________ ml

C6 4

Total ____________ ml
Solid ____________ ml
Liquid ____________ ml

C6 5

Total ____________ ml
Solid ____________ ml
Liquid ____________ ml

70) Decant the centrifuged liquid to a preweighed graduated cylinder. Record the mass and volume of liquid in the graduated cylinder.

C6 1

Total ____________ g
Grad ____________ g
Liquid ____________ g
Liquid ____________ ml

C6 2

Total ____________ g
Grad ____________ g
Liquid ____________ g
Liquid ____________ ml

C6 3

Total ____________ g
Grad ____________ g
Liquid ____________ g
Liquid ____________ ml

C6 4

Total ____________ g
Grad ____________ g
Liquid ____________ g
Liquid ____________ ml

C6 5

Total ____________ g
Grad ____________ g
Liquid ____________ g
Liquid ____________ ml

See step 3
PRB 4/24/00

71) Based on the results from C6 1 through C6 5, determine the amount of water on a wt% basis to evaporate from the three samples to reach the target solids concentrations. Attach the calculations to this Test Plan.

See attached

72) Weight three appropriately sized glass graduated jars or cylinders labeled **C6 EVAP A, C6 EVAP B,** and **C6 EVAP C**.

	C104 5 ~~C6 EVAP A~~ 8915 PRB 5/3/00	C104 15 C6 EVAP B	C104 25-1 C6 EVAP C	C104 25-2
Tare	~~133.1263~~ g	134.2137 ~~1442~~ g	134.2422 g	133.9558 g
Target Slurry mass	102.69 ~~69.6~~ g	68.1 g	68.0 g	68.2 g
Target total mass	236.5815 ~~202.7~~ g	202.3 g	202.2 g	202.2 g

attachment to Test plan 29953-079 PRB 5/31/00

Battelle
Pacific Northwest Laboratories

ENGINEERING WORKSHEET

Prepared By: Paul Bredt	Date: 4/24/00	Project: BNFL 29953

Subject: C-104 Evaporation under step 71 of Test Plan 29953-073

Samples were prepared under BNFL-TI-29953-79 Rev. 1 addendum 1

Solids content is 20 wt% under Test plan 29953-79 Rev. 1

~~5 wt% sample:~~

~~current mass = 17.4 g~~

~~$C_1 M_1 = C_2 M_2$~~

~~$5 \cdot x = 17.4 \cdot 20$~~

~~$x = 69.6\,g$~~ ← New mass target

sample spilled 5/1/00 PRB

15 wt% sample:

current mass = 51.1 g

$$x = \frac{51.1 \cdot 20}{15}$$

$$x = 68.1\,g$$

25-1 25 wt% sample:

Current mass = 85.0 g

$$x = \frac{85.0 \cdot 20}{25} = 68.0\,g$$

25-2 25 wt% sample:

Current mass = 85.2 g

$$x = \frac{85.2 \cdot 20}{25} = 68.2\,g$$

---

5 wt% sample

current mass = 25.6715

$$x = \frac{25.6715 \cdot \cancel{8}20}{5} = 102.69\,g$$

~~73) Measure the distance between the highest and lowest graduation on the graduated glassware using a ruler.~~

~~High_________ ml~~

~~Low_________ ml~~

~~Distance_________ cm~~

See step 10 PRB 4/24/00

"Rhol: Sample C1045" spilled so new sample transferred from "C-104 Remainder". New sample placed in "C104 5" on 5/1/00

74) Transfer the required mass of material into the three graduated ~~glassware~~. glass Jars

C1045 ~~C6 EVAP A~~		C10415 ~~C6 EVAP B~~		C10425-1 ~~C6 EVAP C~~		C10425-2
Total	159.563 g	Total	239.36 g	Total	239.55 g	238.79 g
Tare	~~133.12163~~ 133.8915 g PRB 5/1/00	Tare	134.2137 g	Tare	134.2422 g	133.9558 g
Slurry	~~25.6715~~ g	Slurry	105.146 g	Slurry	105.308 g	104.834 g

These masses include water used to rinse solids during transfer to new Jars. PRB 4/24/00

75) Apply a vacuum to the samples.

76) Monitor the mass of each sample on a regular basis to assess the rate of evaporation. If necessary to accelerate evaporation, place the jar in an oven at 50°C (±10°C).

C-10415 C6 EVAP A		C104 25-1 ~~C6 EVAP B~~		C104 25-2 ~~C6 EVAP C~~	
Date	5/1/00 @ 11 am	Date	5/1/00 @ 11 am	Date	5/1/00 @ 11 am
Mass	239.36	Mass	239.55	Mass	238.79 ← starting masses
Date	>5.7g 5/1/00 @ 2:30 pm	Date	>4.4g 5/1/00 @ 2:30 pm	Date	}~3g 5/1/00 @ 2:30 pm
Mass	233.67	Mass	235.14	Mass	235.87
Date	5/2/00 @ 8:50	Date	5/2/00 8:50 am	Date	5/2/00 @ 8:50 am
Mass	209.08	Mass	224.2	Mass	228.07
Date	5/2/00 @ 1:40 pm	Date	5/2/00 @ 1:40 pm	Date	5/2/00 @ 1:40 pm
Mass	203.8	Mass	217.9	Mass	220.1
Date	5/2/00 @ 9:30	Date	5/3/00 12:00	Date	5/3/00 12:00
Mass	202.7 Final	Mass	213.3	Mass	212.8
Date		Date	5/3/00 3:20	Date	5/3/00 3:20
Mass		Mass	208.9	Mass	207.1
Date		Date	5/4/00 @ 1 pm	Date	5/4/00 @ 1 pm
Mass		Mass	202.35 Final	Mass	198.15 Final

~~target~~
~~current~~
~~water to add~~
~~water added~~

PR Bredt

attachment to
Test Plan 29953-73
PRB 5/31/00

Water addition to C-104 samples
under step 76

	C104 5	C104 15	C104 25-1	C104 25-2
Current mass Total from step 76	159.563 g	202.7 g	202.35 g	197.307 g 7/11/00 MB
Target mass from step 72	236.5815 g	202.3 g	202.2 g	202.2 g
Required water Addition	77.018 g	g	g	4.893 g
New Mass after water addition	Not reweighed g	g	g	g
Water added	~~77~~ 77.0 g	g	g	g

water weighed into 125 ml Jar and then poured onto sample PRB 5/4/00

No No water Needed PRB 5/4/00

C104 25-1 used for density / settling / Rheology study
C104 25-2 to be reserved for mixing / aging study

C6 EVAP A		C6 EVAP B		C6 EVAP C	
Date		Date		Date	
Mass		Mass		Mass	
Date		Date		Date	
Mass		Mass		Mass	

PRB
5/3/00

77) When the mass of each of the slurries reaches the targets calculated above, remove them from the vacuum and cap the sample. Record the mass.

C6 EVAP A		C6 EVAP B		C6 EVAP C	
Total	g	Total	g	Total	g
Tare	g	Tare	g	Tare	g
Slurry	g	Slurry	g	Slurry	g

78) If the samples are in glass jars, transfer ~8g ~~them~~ to appropriately sized preweiged graduated ~~cylinders~~ centrifuge cones with caps or plugs. Record the mass and volume of material in each of the graduated ~~cylinders~~ cones.

PRB 5/4/00

~~C6A~~ C4-5A ρ: 0.94 g/ml		~~C6B~~ C4-15A ρ: 1.64 g/ml		~~C6C~~ C4-25A	
Total	27.357 g	Total	~~30.5?4~~ 28.115 g	Total	28.478 g
cone ~~Grad~~	19.7650 g	cone ~~Grad~~	19.8039 g	cone ~~Grad~~	19.9053 g
Slurry	7.592 g	Slurry	8.311 g	Slurry	8.573 g
Volume	8.0 ml	Volume	5.05 ml	Volume	7.6 ml
Tare = 19.7650		19.8039		19.4053 ρ: 1.13 g/ml	

← sample C104 25-1 used for this part of testing PRB 5/4/00

mass data looks suspect. use mass data in step 85 PRB 5/11/00

rerun on 5/23/00 See attachment PRB 5/31/00

79) Preweigh 3 teflon coated magnetic stir bars.

A	B	C
g	g	g

80) Preweigh 3 appropriately sized volumetric flasks.

A	B	C
g	g	g

81) Place one stir bar in each of the volumetric flasks and fill to the mark with DI water. Record the mass of the volumetric flasks. Calculate the volume of the stir bars by the displacement of water.

A		B		C	
Total	g	Total	g	Total	g
Flask	( )g	Flask	( )g	Flask	( )g
Bar	( )g	Bar	( )g	Bar	( )g
Water	g	Water	g	Water	g

82) Place stir bar A in graduated cylinder **C6 A**, stir bar B in **C6 B**, and stir bar C in **C6 C**.

PRB
5/3/00

C6 EVAP A		C6 EVAP B		~~C6 EVAP C~~	
Date		Date		~~Date~~	
Mass		Mass		Mass	
Date		Date		Date	
Mass		~~Mass~~		Mass	

PRB
5/3/00

77) When the mass of each of the slurries reaches the targets calculated above, remove them from the vacuum and cap the sample. Record the mass.

C6 EVAP A		C6 EVAP B		C6 EVAP C	
Total	g	~~Total~~	g	Total	g
Tare	g	Tare	g	Tare	g
Slurry	g	Slurry	g	Slurry	g

78) If the samples are in glass jars, transfer ~8g ~~them~~ to appropriately sized preweiged graduated ~~cylinders~~ centrifuge cones with caps or plugs. Record the mass and volume of material in each of the graduated ~~cylinders~~ cones. PRB 5/3/00

~~C6 A~~ C4-5 B ρ = 1.39		~~C6 B~~ C4-15 B ρ = 1.67 g/ml		~~C6 C~~ C4-25 B ρ = 1.15 g/ml	
Total	30.161 g	Total	27.806 g	Total	27.896 g
Grad	19.9815 g	Grad	19.5528 g	Grad	20.0465 g
Slurry	10.1795 g	Slurry	8.2532 g	Slurry	7.8495 g
Volume	7.3 ml	Volume	4.95 ml	Volume	6.8 ml
tare	19.9815		19.5538		20.0465

mass data looks suspect. use mass data from step 85 PRB 5/11/00
Density redone on 5/23/00 see attachment PRB

79) Preweigh 3 teflon coated magnetic stir bars.

A	B	C
g	g	g

80) Preweigh 3 appropriately sized volumetric flasks.

A	B	C
g	g	g

81) Place one stir bar in each of the volumetric flasks and fill to the mark with DI water. Record the mass of the volumetric flasks. Calculate the volume of the stir bars by the displacement of water.

A		B		C	
Total	g	Total	g	Total	g
Flask	( )g	Flask	( )g	Flask	( )g
Bar	( )g	Bar	( )g	Bar	( )g
Water	g	Water	g	Water	g

82) Place stir bar A in graduated cylinder **C6 A**, stir bar B in **C6 B**, and stir bar C in **C6 C**.

PRB
5/3/00

attachment to
Test plan 29953-077
PRB Page ______ of ______
5/31/00

Battelle
Pacific Northwest Laboratories

ENGINEERING WORKSHEET

Prepared By: Paul Bredt | Date: 5/22/00 ~~5/23/00~~ | Project: BNFL Melter Feed

Subject: Density of C-104 Melter Feed (No glass formers or 2nd waste products)

C-104 Initial ~~Melter~~ Feed density rerun

	Cone Mass (g)	total w/sample (g)	sample (g)	Ambient volume (ml)	50°C volume (ml)
5 A	4.78	10.04	5.26	5.3	5.3
5 B	4.83	10.35	5.52	~~5.7~~ 5.6	~~5.7~~ 5.6 PRB 5/23/00
15 A	4.75	10.27	5.52	5.3	5.4
15 B	4.81	10.00	5.19	4.9	4.9
25 A	4.81	11.21 ~~6.~~	PRB 5/23/00 ~~5.8~~ 6.4	5.8	5.9
25 B	4.83	11.01	6.18	5.4	5.5

Loaded into cones on 5/23/00
Read Ambient when cell temp was 34°C @ 2:00pm on 5/23/00

	ambient ρ (g/ml)	50°C ρ (g/ml)
5A	0.993	0.993
5B	0.986	0.986
15A	1.04	1.02
15B	1.06	1.06
25A	1.10	1.08
25B	1.14	1.12

Samples placed in 50°C water Bath at 2:30pm
Measured volume of 50°C samples @ 4:00pm

83) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

C45A ~~C6A~~ Time 11:10 am Date 5/8/00
C415A C6 B Time ↓ Date ↓
C425A C6 C Time ↓ Date ↓

84) After turning off the stirrer, record the ambient temperature volume of settled solids layers and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day. Collect video during the first 3 days of settling.

Ambient 30.0 °C

C45A ~~C6A~~ without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)	
10 ~~5~~ min	5/8/00	11:~~15~~20	8.0	7.8		PRB 5/8/00
15 min	↓	11:25	↓	7.7		
25 min		11:35		7.7		
35 min		11:45		7.6		
45 min		11:55		7.6		
55 min		12:05		7.6		
1 hour		12:10		7.5		
1.5 hour		12:40		7.4		← hazy from 7.7 – 7.5
2 hour		~~12~~ 1:10		7.35		
2.5 hour		1:40		7.35		
3 hour		2:10		~7.3		← hazy
3.5 hour		2:40		7.1		← Hazy from 7.0 – 7.5
4 hour		3:10		7.0		← Top of Haze 7.5
4.5 hour	↓	3:40		7.0		haze top 7.5
~~5 hour~~						missed PRB 5/9/00
~~5.5 hour~~						
~~6 hour~~						
~~6.5 hour~~						
~~7 hour~~						
24 hour	5/9/00	8:50		5.0		cloudy to 6.5
28 hour	↓	11:50		5.0		
32 hour		3:30		4.5		← cloudy to 6.5
48 hour	5/10/00	7:50		4.0		
52 hour		4:00	↓	4.0		← cloudy to 5.0
56 hour						

using Glass centrifuge cones for all 6 of the C-1044-1 CS PRB 5/31/00

4/8 = 50% vol

83) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

C45B ~~C6A~~ Time 11:10 am Date 5/8/00

C415B C6 B Time ______ Date ______

C425B C6 C Time ______ Date ______

84) After turning off the stirrer, record the ambient temperature volume of settled solids layers and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day. Collect video during the first 3 days of settling.

Ambient 30.0 °C

~~C415A~~ C45B.

~~C6A~~ without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml) sht	Liquid (ml)
10 ~~8~~ min	5/8/00	11:~~15~~20	~~7.3~~ 7.3	~~7.0~~ 7.0	
15 min		11:25	7.3	7.0	
25 min		11:35		6.9	
35 min		11:45		6.9	
45 min		11:55		6.9	
55 min		12:05		6.85	
1 hour		12:10		6.85	
1.5 hour		12:40		6.8	
2 hour		1:10		6.7	
2.5 hour		1:40		6.7	
3 hour		2:10		6.6	
3.5 hour		2:40		6.5	
4 hour		3:10		6.5	
4.5 hour		3:40		6.5	
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour	5/9/00	8:50		~~5.0~~ 4.8	
28 hour	5	11:50		4.8	
32 hour		3:30		4.5	
48 hour	5/10/00	7:55		4.0	
52 hour		4:00		3.5	
56 hour					

missed PRB 5/8/00

PRB 5/8/00

← haze from ~6.9 to 6.8

← from 6.5–6.9 haze

← top of haze 6.9

top: 6.8

~~top 6.5~~

cloudy to ~~6.5 ml~~ 6.3 ml PRB 5/8/00

← cloudy to 6.0

$\frac{3.5}{7.3} = 47.9$ vol%

~~C425A~~ C4 15A

~~C6-B~~ without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	5/8/00	11:15	5.05	5.05	
15 min		11:25		5.0	
25 min		11:35		5.0	
35 min		11:45		4.9	
45 min		11:55		4.9	
55 min		12:~~00~~05		4.9	
1 hour		12:10		4.9	
1.5 hour		12:40		4.8	
2 hour		1:10		4.7	
2.5 hour		1:40		4.7	
3 hour		2:10		4.6	
3.5 hour		2:40		4.5	
4 hour		3:10		4.5	
4.5 hour		3:40		4.4	
~~5 hour~~					
~~5.5 hour~~					
~~6 hour~~					
~~6.5 hour~~					
~~7 hour~~					
24 hour	5/9/00	8:50		3.6	
28 hour		11:50		3.5	
32 hour		3:30		3.4	
48 hour	5/10/00	7:55		3.3	
52 hour		4:00		3.4	
56 hour					

missed PRB 5/8/00

solution is not hazy

$$\frac{3.4}{5.05} = 67.3 \text{ vol\%}$$

C415B
~~C6 B~~ without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	5/8/00	11:15	4.95	4.95	
15 min		11:25		4.9	
25 min		11:35		4.8	
35 min		11:45		4.75	
45 min		11:55		4.75	
55 min		12:05		4.75	
1 hour		12:10		4.7	
1.5 hour		12:40		4.6	
2 hour		~~12~~ 1:10		4.5	
2.5 hour		1:40		4.5	
3 hour		2:10		4.4	
3.5 hour		2:40		4.4	
4 hour		3:10		4.3	
4.5 hour		3:40		4.3	
~~5 hour~~					
~~5.5 hour~~					
~~6 hour~~					
~~6.5 hour~~					
~~7 hour~~					
24 hour	5/9/00	8:50		3.5	
28 hour		11:50		3.5	
32 hour		3:30		3.4	
48 hour	5/10/00	7:55		3.4	
52 hour		4:00		3.4	
56 hour					

missed PRB 5/8/00

Solution is Not Hazy

$$\frac{3.4}{4.95} = 68.7\%$$

C425A

~~C6C~~ without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	5/8/00	11:15	7.6*	7.6	
15 min	↓	11:25	↓	7.6	
25 min		11:35		7.6	
35 min		11:45		7.6	
45 min		11:55		7.6	
55 min		12:05		7.6	
1 hour		12:10		7.5	
1.5 hour		12:40		7.5	
2 hour		1:10		7.5	
2.5 hour		1:40		7.5	
3 hour		2:10		7.4	
3.5 hour		2:40		7.3	
4 hour		3:10		7.3	
4.5 hour	↓	3:40		7.2	
~~5 hour~~					
~~5.5 hour~~					
~~6 hour~~					
~~6.5 hour~~					
~~7 hour~~					
24 hour	5/9/00	8:50		7.1	
28 hour	↓	11:50		7.1	
32 hour		3:30		7.1	
48 hour	5/10/00	7:55		7.0	
52 hour	↓	4:00	↓	7.0	
56 hour					

missed PRB 5/8/00

on 25A1B
only ~ 0.1 ml of standing liquid before starting settling study. This was after ~5 days of settling. PRB 5/8/00

7.6 ml total with some material smeared on side PRB 5/8/00

85) Place the graduated cylinders in an oven at 50°C (±5°C)for 1 day. Remove the samples and record the mass and volume.

~~C6A~~ C45A

Total 27.357 g
Tare 19.7650 g
Slurry 7.592 g
Volume 7.9 ml
ρ = 0.96 g/ml

C415A
~~C6B~~

Total ~~27.845~~ 24.845 g
Tare 19.8039 g
Slurry ~~8.0411~~ 5.0411 g
Volume 4.8 ml
ρ = 1.05 g/ml

C425A
~~C6C~~

Total 28.457 g
Tare 19.9053 g
Slurry 8.5517 g
Volume 7.8 ml
ρ = 1.00 g/ml

volumes on ~~5/15/00~~ 5/11/00

C425 B
~~C6C~~ without glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min	5/8/00	11:15	6.9*	6.9	
15 min		11:25	6.9	6.9	
25 min		11:35	6.9	6.9	
35 min		11:45	6.8	6.8*	
45 min		11:55	6.8	6.8	
55 min		12:05	6.8	6.8	
1 hour		12:10	6.8	6.75	
1.5 hour		12:40		6.7	
2 hour		~~12~~ 1:10		6.7	
2.5 hour		1:40		6.7	
3 hour		2:10		6.6	
3.5 hour		2:40		6.6	
4 hour		3:10		6.6	
4.5 hour		3:40		6.6	
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour	5/9/00	8:50		6.5	
28 hour		11:50		6.5	
32 hour		3:30		6.5	
48 hour	5/10/00	7:55		6.5	
52 hour		4:00		6.4	
56 hour					

missed 5/8/00 PRB

total volume 6.9 ml with some material smeared on sides PRB 5/8/00

some standing liquid now visible

85) Place the graduated cylinders in an oven at 50°C (±5°C)for 1 day. Remove the samples and record the mass and volume.

C45B
~~C6A~~
Total ~~30.161~~ 26.912 g
Tare 19.9815 g
Slurry ~~10.1795~~ g
Volume 7.25 ml
ρ = 0.96 g/ml
6.9305 g

C415B
~~C6B~~
Total ~~27.806~~ 24.404 g
Tare 19.5528 g
Slurry ~~8.2532~~ g
Volume 4.75 ml
ρ = 1.02 g/ml
4.8512 g

C425B
~~C6C~~
Total ~~27.896~~ 27.844 g
Tare 20.0465 g
Slurry ~~7.8495~~ g ← 7.7975 g
Volume 6.6 ml
ρ = 1.18 g/ml

Volumes on 5/11/00 PRB

86) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

C45A ~~C6A~~ Time 8:45 Date 5/11/00

C415A C6 B Time ↓ Date ↓

C475A C6 C Time ↓ Date ↓

87) Return the graduated cylinders to the oven at 50°C.

88) After turning off the stirrer, record the volume of settled solids layers and liquids every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

C45A
~~C6A~~ without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min	5/11/00	9:15-9:30	7.9	7.9	
60 min		9:45	7.9	7.8	
90 min		10:15	7.9	7.8	
120 min		10:45	7.9	7.8	
150 min		11:15		7.8	
180 min		11:45			
210 ~~hour~~ min		12:15			
240 ~~hour~~ min		12:45			
270 ~~hour~~ min		1:15	7.9	7.8	
300 ~~hour~~ min		1:45	7.9	7.75	
24 hour					
28 hour					
32 hour	5/12/00	4:30	7.8	0.5	
48 hour					
52 hour					
56 hour	5/15/00	9am	~~0.6~~ 7.6	0.6	

Time	total	Solid
2:45	7.9	7.75
3:45	7.9	7.70

← cloudy to 6.2 (32 hour)

← cloudy to 2.5 ml (56 hour)

PRB
5/15/00

86) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

C45B ~~C6A~~ Time 8:45 Date 5/11/00

C415B ~~C6B~~ Time ______ Date ______

C425B ~~C6C~~ Time ______ Date ______

87) Return the graduated cylinders to the oven at 50°C.

88) After turning off the stirrer, record the volume of settled solids layers and liquids every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

C45B PRB 5/3/00

~~C6A~~ without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min	5/11/00	~9:15	7.25	7.25	
60 min		9:45	7.25	7.25	
90 min		10:15	7.25	7.00	
120 min		10:45	7.25	6.80	
150 min		11:15	7.25	6.7	
180 min		11:45			
210 ~~hour~~ m		12:15			
240 ~~hour~~ m					
270 ~~hour~~ m		1:15	7.25	6.65	
300 ~~hour~~ m		1:45	7.25	6.65	
24 hour					
28 hour					
32 hour	5/12/00	4:30	7.2	~0.9	
48 hour					
52 hour					
56 hour	5/15/00	9am	~~0.55~~ 6.8	0.55	

Time	total	Solid
2:45	7.25	6.65
3:45	7.25	6.65

~cloudy to 5.8

← cloudy to 2.5

PRB 5/15/00

C415A
~~C6B~~ without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min	5/11/00	~9:15-9:30	4.8	4.6	
60 min		9:45	4.8	4.5	
90 min		10:15	4.8	4.5	
120 min		10:45	4.8	4.4	
150 min		10:45	4.8	4.3	
180 min					
~~210 hour~~					
~~240 hour~~					
~~270 hour~~		1:15	4.8	4.1	
~~300 hour~~		1:45	4.8	4.0	
24 hour					
28 hour					
32 hour	5/12/00	4:30	4.8	3.5	
48 hour					
52 hour					
56 hour	5/15/00	9am	~~3.5~~ ~~4.6~~ 4.7 PRB 5/15/00	3.5	

time	total	solid
2:45	4.8	3.8
3:45	4.8	3.75

C425A
~~C6C~~ without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min	5/11/00	~9:15-9:30	7.8	7.8	
60 min		9:45	7.8	7.8	
90 min		10:15	7.8	7.75	
120 min		10:45	7.8	7.75	
150 min		11:15	7.8	7.60	
180 min					
~~210 hour~~					
~~240 hour~~					
~~270 hour~~		1:15	7.8	7.5	
~~300 hour~~		1:45	7.8	7.5	
24 hour					
28 hour					
32 hour	5/12/00	4:30	7.7	7.3	
48 hour					
52 hour					
56 hour	5/15/00	9am	7.4	7.4	

time	total	solid
2:45	7.8	7.45
3:45	7.8	7.45

C415B
~~C6 B~~ without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min	5/11/00	~9:15-9:30	4.75	4.65	
60 min		9:45	4.75	4.60	
90 min		10:15	4.75	4.60	
120 min		10:45	4.75	4.50	
150 min		11:15	4.75	4.40	
180 min					
210 ~~hour~~					
240 ~~hour~~					
270 ~~hour~~		1:15	4.75	4.2	
300 ~~hour~~		1:15	4.75	4.2	
24 hour					
28 hour					
32 hour	5/12/00	4:30	4.7	3.7	
48 hour					
52 hour					
56 hour	5/15/00	9Am	4.6	3.8	

Time	total	Solid
2:45	4.75	4.1
3:45	4.75	3.9

C425B
~~C6 C~~ without glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min	5/11/00	~9:15-9:30	6.6	6.6	
60 min		9:45	6.6	6.6	
90 min		10:15	6.6	6.55	
120 min		10:45	6.6	6.55	
150 min		11:45		6.50	
180 min					
210 ~~hour~~					
240 ~~hour~~					
270 ~~hour~~		1:15	6.6	6.50	
300 ~~hour~~		1:15	6.6	6.50	
24 hour					
28 hour					
32 hour	5/12/00	4:30	6.6	6.6	
48 hour					
52 hour					
56 hour	5/15/00	9am	6.5	6.5	

time	total	Solid
2:45	6.6	6.45
3:45	6.6	6.45

89) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to 1000 s^{-1}. Print out a copy of the rheogram and attach to this test plan.

Viscometer M5 Location A-Cell South Geometry MV I

Viscosity 95.5 cP Lot 111199 Manufacturer Brookfield

File name 050200A B C Date analyzed 5/2/00

90) Remove the graduated cylinders from the oven and allow to cool overnight.

91) While stirring the samples on a magnetic stir plate remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to 300 s^{-1} over 2 minutes and then returning from 300 to 0s^{-1} over 2 minutes Print out a copy of the rheograms and attach to this test plan.

	File name (25°C)	Date analyzed	50°C
C6 A	~~0504~~ 051200A	5/12/00	051200 H
C6 A Duplicate	051200B		051200 I
C6 B	051200 C		051200 J
C6 B Duplicate	051200D		051200 K
C6 C	051200 E		Insufficient material
C6 C Duplicate	051200 F 051200 G		

actually AZ-102 Samples under st. 28 PRB 5/31/00

92) Return this material to the respective graduated cylinders.

material in Rheometer + cones returned to respective jars and supernatant decanted to return samples to target weight.
See attachment
PRB
5/31/00

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ENGINEERING WORKSHEET

Prepared By: Paul Bredt | Date: 5/22/00 | Project: BNFL Melter ~~Field~~ Feed

Subject:

Mass of Recovered samples following Rheology

Sample	Target Total mass with jar	total mass ~~target~~	water to add	Final mass
C104 5wt%	236.5815	203.11	33.47	236.61
15wt%	202.3	182.00	20.30	202.54
25wt%-1	202.2	197.28	4.92	202.20
AZ-102 5wt%	214.41	166.26	48.15	214.46
15wt%	211.75	209.43	2.32	211.73
25wt%-1	~~269.59~~ 282.62	~~286.41~~ 271.176	~~-16.82~~	282.774

adjusted to 20wt% as per email with Stuart Arm
PRB
5/31/00

attachment to test plan 29953-073
PRB
5/31/00

Balance check 1000g = 1000.00g reading on A-Cell North

DOE-RL Richland, WA ☆U.S. GPO: 1997 - 592-715 54-1007-114 (1/83)

15 min due to stirrer imbalance & glass jars

93) Add the BNFL prescribed quantity of intermediate waste products to each of the three graduated cylinders and stir for ~~1 hour~~. Use the space below to record the quantity of intermediate waste products added and the time and date stirring started and stopped.

C6 A Stir Start (time/date) 2:30 Stir Stop (time/date) 2:45
5 wt + 50

Source Chemical	Target wt%	Amount (g)	Actual Added (g)
Sr/TRU precipitate		6.149 ~~1.018 g~~	5.484
CS Eluant		0.6 ml	0.737 g

Lost 0.365 H_2O

St wt
235.757 g
after stirring
Sr/TRU
241.254 g
after CS
241.971 g
after stirring
240.811 g
added ~2.02 g H_2O
242.805 g
Final

C6 B Stir Start (time/date) 3:05 Stir Stop (time/date) 3.30
15 wt 50

Source Chemical	Target wt%	Amount (g)	Actual Added (g)
Sr/TRU precipitate		12.231 g	12.062 g
CS Eluant		1.1 ml	1.436 g

Lost H_2O 0.773

St wt
201.770
Sr/TRU
213.832 g
CS Eluant
215.268 g
After Stirring
213.795 g
Add ~2.243 H_2O
216.532 g
final

I'm short -
Target was
216.038

05/26/00

C6 C Stir Start (time/date) 07:56 Stir Stop (time/date) 7:35

25 min+ So

Lost 1.135g H_2O

Source Chemical	Target wt%	Amount (g)	Actual Added (g)
Sr/tru			20.160g
Cs Eluent		1.9 mL	2.451g

start wt
201.065

after adding Sr/TRU
221.225

after Cs Eluent
223.676

After stir
222.045

after H_2O
224.758

~~94) Cap the graduated cylinders and record the mass and volume.~~

	C6 A		**C6 B**		**C6 C**
Total	______ g	Total	______ g	Total	______ g
Tare	______ g	Tare	______ g	Tare	______ g
Slurry	______ g	Slurry	______ g	Slurry	______ g
Volume	______ ml	Volume	______ ml	Volume	______ ml

~~95) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.~~

C6 A Time ______ Date ______

C6 B Time ______ Date ______

C6 C Time ______ Date ______

Test run in 15 ml centrifuge cones
See attached sheet
PRB
5/30/00

Attachment to Test
29953-073 Page ____ of ____

ratories

ENGINEERING WORKSHEET

Bredt	Date: 5/23/00	Project: BNFL HLW Melter Feed

/TRU simulant

/TRU was prepared by Rich Hallen under TI-082
tle Number 1 was used for this work

removed 2 subsamples for drying test on 5/23/00
sig Balance ~~388-06-01-018 exp. 8/00~~
362-06-01-041 exp 2/01

Bottle 1	Bottle 2
15.9443 g	16.8963 g
13.2983	13.2878 g
2.6460 g ✓	
~~14.0050~~ PRB 5/24/00 g	3.6085 g ✓

laced in oven at 105°C indicated on mercury thermometer
at 850 am on 5/23/00

Mass @ 2:40pm on 5/23/00

1 14.0064 g

2 14.2590

Mass @ 9:30am on 5/24/00

1 14.0050 − 13.2983 = 0.7067 g ✓

2 14.2559 − 13.2878 = 0.9681 g ✓

$\frac{0.7067}{2.646} = 0.267 \frac{\text{g solids}}{\text{g sludge}}$ ✓ $\frac{0.9681 \text{ g}}{3.6085} = 0.268 \frac{\text{g solids}}{\text{g sludge}}$ ✓

$\boxed{\bar{x} = 0.268 \text{ g solids/g sludge}}$ ✓

	5 wt%	15 wt%	25 wt%-1	75 wt%-:
olids added	1.648	3.278	5.456	5.472
solids added	6.149	12.231	20.358	20.418
	✓	✓	✓	

hecked by LKJagoda 5/24/00 ☆U.S. GPO: 1997 - 592-715 [signature] 5/24/00 54-1007-114

Mass of Sr/TRU simulants for C-104 Testing

5wt% Syringe 1 tare 11.1101 11.805g

sample 6.138g wt after empty

15wt%. Syringe 2 tare 11.6525g

sample 9.8115g 2.420

Syringe 3 tare 11.6717g

Sample 2.4440g total $\boxed{12.256}$

25-1 wt%

Syringe 4 tare 11.6547g

sample 10.3147g 10.0433

Syringe 5 tare 11.6828g

Sample 10.0012g total $\boxed{20.316}$

25-2 wt%

Syringe 6 tare 11.6086g

Sample 10.4267g Total: 22.0353g

after adding T= 11.64g

Syringe 7 tare 11.7415g 9.9913

Sample 10.0566g total 21.7981g

after adding T=11.75g total $\boxed{20.4833}$

attachment to Test Plan 29953-073

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ENGINEERING WORKSHEET

Prepared By: LK Jagoda | Date: 5/26/00 | Project: BNFL-TP-29953-073

Subject: C-104 & AZ-102 Settling Cones
w/secondary waste; w/secondary waste & Glass formers

	Tare(g)	Filled(g)	Sample	Gross wt on 6-9-00	6-9-00
AZ-5-A	4.506	12.832	8.326	12.822	12.816
AZ5-B	4.508	12.386 ~~12.858~~	7.878	12.375	12.370
AZ-15-A	4.509	12.858	8.349	Not	12.840
AZ-15-B	4.512	12.744	8.232	Measured	12.726

AZ 5 Bottle Before 225.936 g After 209.451 g Sample 16.204 Lost 0.281 g
AZ 15 Bottle Before 244.460 g After 227.345 g. sample 18.581 Lost 0.534 g

	Tare	Filled	Sample	Gross wt on 6-9-00	6-9-00
C-5A	4.445	11.837	7.392	11.826	11.820
C-5B	4.820	12.523	7.703	12.517	12.509
C-15A	4.470	11.756	7.286 ← 7.286 RB 5/30/00	11.746	11.739
C-15B	4.784	12.128	7.344	12.062 - adj - add H$_2$O to 12.107	12.066
C-25A	4.389	11.902	7.513	Not measured	11.855
C-25B	4.792	11.580	6.788	not measured	11.556

C-104 5 Bottle Start 242.697 g After 227.513 g Sample 15.095 g Lost 0.089 g
C-104-15 Bottle Start 216.365 g After 201.429 g Sample 14.63 g Lost 0.306 g
C-104-25 Bottle Start 224.707 g After 209.483 g Sample 14.301 g Lost 0.923 g

5/26/00

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ENGINEERING WORKSHEET

Prepared By:	Date:	Project:
Paul Bredt	5/30/00	Attachment to BNFL TP 29953-073

Title/Subject: Settling study C-104 w/secondary waste + AZ-102 w/Glass formers

- Reference to step 94 of test plan
- Glass formers / secondary wastes were added 5/26/00, so samples have settled in cones for ~4 days.
- AZ-102 15wt% samples have shown very little settling, and C-104 25wt% samples have shown none

Measured volumes @ 9:15 am on 5/26/00

		Total (ml)	Solids (ml)	mass (g)	density (g/ml)
C104	5A	7.2	~~4.6~~ 1.7	7.392	1.03
	5B	7.5	1.8	7.703	1.03
	15A	6.4	5.3	7.286	1.14
	15B	6.8 after stirring 6.5	5.6F lots of gass in solids	7.344	1.08 after stirring 1.13
	25A	6.9	6.9	7.513	1.09
	25B	6.7	6.7	6.788	1.01
AZ102	5A	~~7.7~~ 7.65	~~4.6~~ 4.45	8.326	1.09
	5B	7.2	4.3	7.878	1.09
	15A	6.6	6.35	8.349	1.26
	15B	6.7	6.45	8.232	1.23

- No meniscus on AZ-102, but ~0.2ml of meniscus on C-104 samples
- C-104 read on Bottom of meniscus
- Gas bubbles seen in C-104 15B, No bubbles in A
- C-104 results are suggesting of trapped gas

Prepared By: Paul Bredt	Date: 5/30/00	Project: Attachment to TP 29953-073

Subject: settling study C-104 w/secondary waste & AZ-102 w/glass formers

- samples C-104 ~~15~~ 25 A & B did not settle over 4 days, so no included in this study
- Samples AZ-102 15 A & B did not settle, so not included in this study

C-104 ↓ ↙AZ-102

• Ambient study steps 96 & step 45

C-104 shaken @ 10:35 2am

ate	Time	5A Total	solids	~~4~~5B total	solids	15A Total	solids	15B total	Solids
/30	10:35	7.5	7.3 liquid is	7.8	7.4 liquid is	~~7.1~~ 6.6	~~7.1~~ 6.6	6.7	6.7
/30	10:45	7.~~4~~5	7.1 cloudy	7.7	7.2 cloudy	6.5	6.3	6.7	6.3
	11:00	7.5	4.3	7.7	4.6	6.5	6.3	6.7	6.3
	11:10	7.5	3.4	7.7	3.6	6.6	6.2	6.7	6.2
	11:20	7.5	2.7	7.7	2.9	6.6	6.2	6.7	6.2
	11:30	7.2	2.2	7.5	2.4	6.6	6.1	6.6	6.2
	12	7.2	2.0	7.5	2.2	6.6	6.0	6.5	6.1
	12:30	7.2	1.9	7.5	2.0	6.6	6.0	6.5	6.0
	1	7.2	~~1.8~~ 1.9	7.5	2.0	6.6	5.8	6.5	5.8
	1:30	7.2	1.8	7.6	1.9	6.5	5.7	6.5	5.[illegible]
	2	7.2	1.75	7.6	1.9	6.5	5.6	6.5	5.7
	2:30	7.2	1.75	7.6	1.9	6.5	5.6	6.5	5.7
	3	7.2	1.75	7.6	1.9	6.5	5.55	6.5	5.6
	3:30	7.2	1.75	7.6	1.8	6.5	5.5	6.5	5.6
	4	7.2	1.7	7.6	1.75	6.5	5.5	6.5	5.6
31/00	7:30 am	7.3	1.6	7.5	1.7	6.5	5.2	6.5	5.3
	2:00 pm	7.3	1.6	7.5	1.6	6.5	5.1	6.5	5.2
/1	8:00 am	7.3	1.5	7.5	1.55	6.5	5.1	6.5	5.2
/2	8:00 am	7.3	1.45	7.5	1.5	6.5	5.0	6.5	5.1

used plastic centrifuge cones

Battelle
Pacific Northwest Laboratories

ENGINEERING WORKSHEET

Prepared By: Paul Bredt | Date: 5/30/00 | Project: Attachment to TP 29953-073

Subject: Settling Study

8/10/00

AZ-102 - Ambient (with PRB glass formers)

shaken @ ~~10:35~~ 10:32 am

Date	Time	AZ-102 5A total	solids	5B total	solids
5/30/00	~~10:30~~ 10:35	7.6	7.3	7.3	7.1
5/30/00	10:45	7.6	7.2	7.3	6.7
	11:00	7.6.	7.0	7.3	6.5
	11:10	7.6	6.8	7.3	6.3
	11:20	7.6	6.5	7.3	6.0
	11:30	7.5	6.4	7.2	5.8
	12	7.5	6.0	7.2	5.5
	12:30	7.5	5.5	7.2	5.2
	1	7.5	5.3	7.2	5.0
	1:30	7.5	5.0	7.1	4.8
	2	7.5	4.9	7.1	4.7
	2:30	7.5	4.8	7.1	4.6
	3	7.5	4.65	7.1	4.5
	3:30	7.5	4.5	7.1	4.4
	4	7.5	4.5	7.1	4.4
5/31/00	7:30 am	7.5	4.2	7.1	4.2
	2:00 pm	7.5	4.1	7.1	4.1
6/1/00	8:00 am	7.5	3.8	7.1	3.9
6/2/00	8 am	7.5	3.7	7.1	3.8

used plastic centrifuge cones that were 3.5 cm from 4 to 10 ml

$\frac{3.5\ cm}{6\ ml} = 0.583\ \frac{cm}{ml}$

PRB
6/2/00

AZ-102 with Glass formers and C-104 with Secondary Waste Products

6/7/00 Start shaking @ 10:05am EN @ →

	AZ-102				C-104							
	5A		5B		5A		5B		15A		15B	
Time	Total	Solids	Total	Solids	Total	Solids	Total	Solids	Total	Solids	Total	Solids
~10:10	~7.6	~7.6	~7.6	~7.6	~7.4		~7.6		~6.5		~6.8	
10:25	7.6	7.3	7.3	6.9	7.5	7.0	7.6	7.0	~6.5	SAME	6.6	SAME
10:50	7.6	6.8	7.3	6.5		6.8		6.4	6.5	~6.4	6.6	~6.5
	~~4th bottom~~	~~~2.5~~	~~bottom~~	~~2.5~~	10:52 bottom	~2.5	10:52 bottom	~2.5				
~~10~~1:40	~7.6	5.85	~7.6	5.5	7.5	2.2	~7.6	2.35	~6.5	6.2	~6.5	6.2
1:40pm	7.6	5.0	7.6	4.7	7.6	2.0	7.8	1.90	6.8	5.85	6.5	5.9
2:45pm	7.6	4.7	7.6	4.6	7.6	1.9	7.7	1.8	6.8	5.8	6.5	5.5
3:35pm	7.6	4.65	7.6	4.50	7.6	1.85	7.7	1.9	6.8	5.7	6.5	5.5
8:30am	7.65	4.60	7.7	4.40	7.5	1.75	7.7	1.7	6.8	5.35	6.5	5.45
10:45am	7.65	4.55	7.7	4.40	7.5	1.75	7.7	1.7	6.8	5.35	6.5	5.15
3:30pm	7.65	4.5		4.40		1.75		1.7		5.30		5.13
8:30	7.65	4.5	7.3	4.4	7.4	1.75	7.6	1.7	6.8	5.3	6.4	5.1

Best vol reading

6/8/00

6/9/00

Attachment to
Test Plan 29953-073

Density of AZ-102 with Glass formers
C-104 with 2nd waste products

Cones were prepared on 5/26/00 and settling studies were conducted at ambient + 50°C. However gas bubbles in sludge made density measurements unreliable.

Samples will be centrifuged @ ~1000 xg for 5 min to remove bubbles and then the volumes will be measured @ ambient 33°C (on readout 2215 exp 10/11/00) and @ 50°C (in an oven at 50° on readout 2013 exp 8/01) for 1 hour

~~Mass~~ Sample		(g) Mass on 6/9/00	Total Volume (ml) at ambient	after 1 hr at 50°C
AZ-102	5A	12.816	7.7 ~~ρ=~~	7.8
	5B	12.370	7.2	7.3
	15A	12.840	6.8	6.8
	15B	12.726	6.75	6.8
C-104	~~3~~5A	11.820	7.3 ~~ρ=1.62 g/ml~~	7.3
	5B	12.509	7.6 ~~ρ=1.65 g/ml~~	7.6
	15A	11.739	6.5 ~~ρ=1.81 g/ml~~	6.5
	15B	12.066	6.6 ~~ρ=1.83 g/ml~~	6.6
	25A	11.855	6.2 ~~ρ=1.91 g/ml~~	6.2
	25B	11.556	~~5.8~~ ~~5.6~~ 5.8 ~~ρ=1.99 g/ml~~ PAB 6/9/00 ignore using gross wts	5.8

performed by
Paul B[illegible]
6/9/00

attachment to Test Plan 29953-73 — in order 6/9/00

Density of settled samples at ambient

AZ-102

5A

$$\frac{12.816 - 4.506}{7.7} = 1.08\ g/ml$$

@ 50°C: 8.31/7.8 = 1.06 g/ml

5B

(12.370 - 4.508)/7.2 = 1.09 g/ml

7.862/7.3 = 1.08 g/ml

15 A

(12.840 - 4.509)/6.8 = 1.23 g/ml

same as ambient

15 B

(12.726 - 4.512)/6.75 = 1.22 g/ml

C-104

5 A

(11.820 - 4.445)/7.3 = 1.01 g/ml

5 B

(12.509 - 4.820)/7.6 = 1.01 g/ml

15 A

(11.739 - 4.470)/6.5 = 1.12 g/ml

15 B

(12.066 - 4.784)/6.6 = 1.10 g/ml

25 A

(11.855 - 4.389)/6.2 = 1.20 g/ml

25 B

(11.556 - 4.792)/5.8 = 1.17 g/ml

96) After turning off the stirrer, record the ambient temperature, volume of settled solids layers and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day. Collect video during the first 3 days of settling.

Ambient_____________°C

C6A with waste products at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

see attachment
PRB
5/31/00

C6 B with waste products at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See attachment
PRB 5/31/00

C6 C with waste products at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See attachment
PRB
5/31/00

97) Place the graduated cylinders in an oven at 50°C (±5°C) for 1 day. Remove the samples and record the mass and volume.

	C6 A		**C6 B**		**C6 C**
Total	______ g	Total	______ g	Total	______ g
Tare	______ g	Tare	______ g	Tare	______ g
Slurry	______ g	Slurry	______ g	Slurry	______ g
Volume	______ ml	Volume	______ ml	Volume	______ ml

98) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

C6 A Time ______ Date ______

C6 B Time ______ Date ______

C6 C Time ______ Date ______

See attachment
8/10/00

99) Return the graduated cylinders to the oven at 50°C.

100) After turning off the stirrer, record the volume of settled solids layers and liquids every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

C6 A with intermediate waste products at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

C6 B with intermediate waste products at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See ~~att~~ attachment 8/10/00

C6 C with intermediate waste products at 50°C

See attachment 8/10/02

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

101) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to approximately 1000 s^{-1}. Print out a copy of the rheogram and attach to this test plan.

Viscometer M5 Location A cell South Geometry MV I

Viscosity 95.5 cP Lot 111199 Manufacturer Brookfield

File name 060100 A B C Date analyzed 6/1/00

102) Remove the graduated cylinders from the oven and allow to cool overnight.

103) While stirring the samples on a magnetic stir plate remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to 300 s^{-1} in 2 minutes and then returning from 300 to 0s^{-1} in 2 minutes. Print out a copy of the rheograms and attach to this test plan.

25°C

C6 A File name ________ Date analyzed

C6 A Duplicate File name ________ Date analyzed

C6 B File name ________ Date analyzed

C6 B Duplicate File name ________ Date analyzed

~~C6 C~~ File name ________ Date analyzed

25°C	50°C
5wt% 060100 D E	5wt% 060200 F G H
15wt% 060100 F G	15wt% 060200 I J
25wt% 060100 H I J K	25wt% 060200 K L M

C6 C ~~Duplicate~~ File name __________ Date analyzed See previous page 8/10/00

104) Return this material to the respective graduated cylinders.

105) Add the BNFL prescribed quantity of glass formers to each of the three graduated cylinders and stir for 1 hour. Use the space below to record the quantity of glass formers added and the time and date stirring started and stopped.

~~C6A~~ C104 5wt% Stir Start (time/date) 9:50 Stir Stop (time/date) 10:50 6/19/00

after mixing added 7.95g of DI H2O PRB on 6/20/00

Glass Formers:

Source Chemical	Manufacturer	Lot#	Target wt%	Amount (g)	Actual Added (g)

See below PRB 6/16/00

weighed out Glass formers from mixture Lynette prepared on 6/15/00

	5wt%	15 wt%	25-1	25-1 2	25-2	25-2 2
Tare	24.4464	24.3132	24.4254	24.1354	24.1967	24.5155
Glass	14.0981	28.0235	27.5406	19.1443	27.5024	19.2739
Total	38.5445	51.3367	51.9660	43.2997	51.6991	43.7894

prior to glass addition, samples were evaporated using a flow of air back to near target masses, will add remaining water later to prevent overflow

	5 wt%	15 wt%	25-1 wt%
Target	242.805	216.~~532~~ 038 PRB	224.758
Final mass	234.930	212.577 PRB 6/19/00	211.520

Glass formulations in this report for AW-101 and AN-107 are considered to be proprietary by VSL/Duratek

C104glass.xls
5/24/00
PR Bredt

C-104 Glass Additive Formulation

102.69/400 = .25673

68.1/400 = .17025

68/400 = 0.17

68.2/400 = 0.1705

Additive	Based on 400 g of slurry 20 wt%	5 wt%	15 wt%	25 wt%	Actual mass 102.69 5 wt%	68.1 15 wt%	68 25 wt%-1	68.2 25 wt%-2
Sr/TRU Precipitate (dry solids basis)	25.674	6.418	19.256	32.092	1.648	3.278	5.456	5.472
Cs Eluant (27 g total solids/ml 100 solution)								
in ml	8.8	2.2	6.6	11	0.6 .57	1.1 1.12	1.9 1.87	1.9 1.88
Na2B4O7□10H2O	58.000	14.500	43.500	72.500	3.723	7.406	12.325	12.361
LiOH□H2O	36.800	9.200	27.600	46.000	2.362	4.699	7.820	7.843
Na2CO3	3.525	0.881	2.644	4.406	0.226	0.450	0.749	0.751
SiO2 (Sil-co-Sil 75)	116.280	29.070	87.210	145.350	7.463	14.848	24.710	24.782
ZnO (KADOX-920)	4.930	1.232	3.698	6.162	0.316	0.630	1.048	1.051
Sugar (Granular)	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Total Glass Formers					14.090	28.032	46.651	46.788

Checked by LK Jagoda

5/23/00

Attachment to BNFL 29953-073

Glass Additive Preparation

attachment to Test Plan BNFL-~~2253~~ 29953-073 PRB 6/16/00

Glass formulations in this report for AW-101 and AN-107 are considered to be proprietary by VSL/Duratek

C104glass.xls
5/24/00
PR Bredt

	Based on 400 g of slurry				Actual mass				Total	Total + 20%	
					102.69	68.1	68	68.2			
Additive	20 wt%	5 wt%	15 wt%	25 wt%	5 wt%	15 wt%	25 wt%-1	25 wt%-2			
Sr/TRU Precipitate (dry solids basis)	25.674	6.418	19.256	32.092	1.648	3.278	5.456	5.472			
Cs Eluant (27 g total solids/ml solution) in **ml**	8.8	2.2	6.6	11	0.565	1.124	1.870	1.876			
Na2B4O710H2O	58.000	14.500	43.500	72.500	3.723	7.406	12.325	12.361	35.815	42.978	Lynette,
LiOHH2O	36.800	9.200	27.600	46.000	2.362	4.699	7.820	7.843	22.724	27.269	please
Na2CO3	3.525	0.881	2.644	4.406	0.226	0.450	0.749	0.751	2.177	2.612	make up a
SiO2 (Sil-co-Sil 75)	116.280	29.070	87.210	145.350	7.463	14.848	24.710	24.782	71.802	86.163	total +
ZnO (KADOX-920)	4.930	1.232	3.698	6.162	0.316	0.630	1.048	1.051	3.044	3.653	20% mix
Sugar (Granular)	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	
Total Glass Formers					14.090	28.032	46.651	46.788	135.561	162.673	

Batch DATA

	Date	Amount Actually Added	
Na2B4O710H2O		42.98	
LiOHH2O		27.27	
Na2CO3		2.61	
SiO2 (Sil-co-Sil 75)		86.16	
ZnO (KADOX-920)		3.65	
Sugar (Granular)		0.00	
Total Glass Formers		182.32	Final Mass
		19.73	Tare
		162.59	Final Mass of Glass Formers

Glass former preparation at APEL by Lynette Jagoda on 6/15/00

6/19/00

C6 B Stir Start (time/date) 10:55 Stir Stop (time/date) 11:55

after mixing added 3.520g of DI H2O 6/20/00 PRB

Glass Formers:

Source Chemical	Manufacturer	Lot#	Target wt%	Amount (g)	Actual Added (g)

See page 50 PRB 6/19/00

6/19/00

C6 C Stir Start (time/date) 1:00 Stir Stop (time/date) 1:50

Glass Formers:

Source Chemical	Manufacturer	Lot#	Target wt%	Amount (g)	Actual Added (g)

See page 50 PRB 6/19/00

added 13.116g H_2O To 25-1 before mixing 6/19/00 PRB

106) Cap the graduated cylinders and record the mass and volume.

C6 A		C6 B		C6 C	
Total	____ g	Total	____ g	Total	____ g
Tare	____ g	Tare	____ g	Tare	____ g
Slurry	____ g	Slurry	____ g	Slurry	____ g
Volume	____ ml	Volume	____ ml	Volume	____ ml

107) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

C6 A Time ____ Date ____ See attachment 8/10/00 / RB

C6 B Time ____ Date ____

C6 C Time ____ Date ____

samples loaded in duplicate into 10 ml plastic centrifuge cones

	C5A	C5B	C15A
Total	13.487 g	13.145 g	14.816 g
Tare	4.469 g	4.503 g	4.445 g
sludge	9.018 g	8.642 g	10.371 g

	C15B	C25A	C25B
Total	15.835 g	16.389 g	16.027 g
tare	4.514 g	4.445 g	4.465 g
sludge	11.321 g	11.944 g	11.562 g

7/6 wt. check
C5A - 13.484 ✓
C5B - 13.148 ✓
C15A - 14.819 ✓
C15B - 15.718 ✓

7/10 wt check
RB
C5A - 13.430 g
C5B - 13.111 g
C15A - 14.781
C15B - 15.594

8/3 wt check
C5A - 13.410 g
C5B - 13.091 g
C15A - 14.761 g
C15B - 15.544 g
C25A - 16.242
C25B - 15.881

7/11/00 - centrifuged all 6 cones @ ~1000xg for 30 min
Then recorded volumes

		ρ (g/ml)			ρ (g/ml)
C5A	8.43 RB 7/11/00 ml	1.09 g/ml	C25A	8.5 ml	1.41
C5B	8.1 ml	1.07	C25B	8.2 ml	1.41
C15A	8.0 ml	1.30			
C15B	8.6 ml	1.33			

8/3/00 · samples placed in oven @ 50°C at 10am
· volumes read at 2pm

C5A 8.3
C5B 8.0
C15A 8.1
C15B 8.6
C25A 8.4
C25B 8.1

C-104 with Secondary Waste Products and Glass Formers

			C-104											
			8.2 5A		7.8 5B		8.1 15A		9.0 15B		25A		25B	
	Date	Time	Total	Solids	Total	Solids	Total	Solids	Total	Solids	Total	Solids	Total	Solids
5 min	6/21/00	9:25 am	8.2	7.2	7.8	6.5	8.1	8.1	9.1	9.1				
15	6/21	9:35	8.2	6.1	7.8	5.3	8.1	8.1	9.1	9.1				
25	6/21	9:45	8.2	4.7	7.8	4.0	8.1	8.1	9.1	9.1				
35	6/21	9:55		3.8		3.3	8.1	8.1	9.1	9.1				
45	6/21	10:05		3.5		3.0								
55	6/21	10:15		3.4		2.9								
1 hr	6/21	10:20		3.3		2.8								
1.5 hr	6/21	10:50		3.0		2.7								
2 hr	6/21	11:20		2.9		2.55	8.1	8.0	9.1	9.0				
2.5	6/21	11:50		2.8		2.50	8.1	7.9	9.1	9.0				
3	6/21	12:20		2.8		2.50								
3.5	6/21	12:50		2.7		2.4								
4		13:20		2.7		2.3								
4.5		13:50		2.6		2.3								
5		14:20		2.6		2.3								
5.5		14:50		2.5		2.3								
6		15:20		2.5		2.3								
6.5		15:50		2.5		2.3								
7		16:20		2.5		2.3		7.8		8.9				
7½		16:50		2.5		2.3								
24	6/22	9:00		2.5		2.3		7.2		8.2				
32				2.4		2.2		7.0		7.8				

would not settle
PAB
8/10/00

Ambient Settling Study

C-104 with Secondary Waste Products and Glass Formers

			C-104											
			5A		5B		15A		15B		25A		25B	
	Date	Time	Total	Solids	Total	Solids	Total	Solids	Total	Solids	Total	Solids	Total	Solids
48				2.3		2.1		6.9		7.6				
52														
56														
	6/26	11:40am	8.3	2.1	7.8	1.9	8.0	6.5	8.8	7.2				

would not settle PRB 8/10/00

record ~30 min first day

50°C Settling Study

Attachment to Test Plan 29953-073

6/27

C-104 with Secondary Waste Products and Glass Formers

			C-104											
Shake @	9:10		8.3 5A		8.0 5B		8.1 15A		9.0 15B		25A		25B	
	Date	Time	Total	Solids	Total	Solids	Total	Solids	Total	Solids	Total	Solids	Total	Solids
30min	9:40	9:40	8.5	4.0	8.1	3.6	8.0	—	~8.8	—				
60	10:12	10:12	8.5	3.3	8.1	3.1	8.0	8.0	8.8	8.65				
90	6/27	10:40		3.1		2.8		7.8		8.6				
120	6/27	11:10		2.9		2.7		7.7		8.5				
~~150~~	6/27													
180	6/27	12:10		2.8		2.7		7.7		8.45				
210	6/27	12:40		2.8		2.6		7.7		8.40				
240	6/27	1:10		2.8		2.55		7.6		8.35				
270	6/27	1:40		2.75		2.55		7.5		8.30				
300	6/27	2:10		2.75		2.55		7.5		8.30				
24hr	6/27	2:40		2.70		2.55		7.5		8.25				
28hr	6/27	3:10		2.70		2.55		7.50		8.25				
32hr	6/27	3:40		2.70		2.55		7.45		8.20				
48hr	6/27	4:10		2.70		2.55		7.45		8.20				
52hr	6/28	8:00		2.70		2.50		7.25		8.00				
60hr	6/28	4:00		2.70		2.45		7.20		7.95				

would not settle 8/10/00 PRB

108) After turning off the stirrer, record the ambient temperature, volume of settled solids layers and liquids after 5 minutes, then every 10 minutes for the first hour, and then every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day. Collect video during the first 3 days of settling.

Ambient____________°C

C6 A with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See attachment

C6 B with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See attachment

C6 C with glass formers at ambient temperature

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
5 min					
15 min					
25 min					
35 min					
45 min					
55 min					
1 hour					
1.5 hour					
2 hour					
2.5 hour					
3 hour					
3.5 hour					
4 hour					
4.5 hour					
5 hour					
5.5 hour					
6 hour					
6.5 hour					
7 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

109) Place the graduated cylinders in an oven at 50°C for 1 day. Remove the samples and record the mass and volume.

C6 A			C6 B			C6 C		
Total	______	g	Total	______	g	Total	______	g
Tare	______	g	Tare	______	g	Tare	______	g
Slurry	______	g	Slurry	______	g	Slurry	______	g
Volume	______	ml	Volume	______	ml	Volume	______	ml

See attachment

110) Mobilize the material in each of the graduated cylinders using a magnetic stir plate. After ~2 minutes, turn off the stir plate and record the time and date for each of the three samples.

C6 A Time ____________ Date____________

C6 B Time ____________ Date____________

C6 C Time ____________ Date____________

111) Return the graduated cylinders to the oven at 50°C.

112) After turning off the stirrer, record the volume of settled solids layers and liquids every half hour until the end of the work day. As well, record these volumes every 4 hours during the second and third day.

C6 A with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

See attachment

C6 B with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

C6 C with glass formers at 50°C

Target	Date	time	Total (ml)	Solid (ml)	Liquid (ml)
30 min					
60 min					
90 min					
120 min					
150 min					
180 min					
210 hour					
240 hour					
270 hour					
300 hour					
24 hour					
28 hour					
32 hour					
48 hour					
52 hour					
56 hour					

see attachment

113) If not performed in the last 30 days, analyze one standard between 10 and 100 cP for shear stress as a function of shear rate at 25°C from 0 to approximately 1000 s^{-1}. Print out a copy of the rheogram and attach to this test plan.

Viscometer M5 Location A cell Geometry MV I

Viscosity 95.5 cP Lot 11 11 99 Manufacturer Brookfield

File name 060100A B C Date analyzed 6/1/00

114) Remove the graduated cylinders from the oven and allow to cool overnight.

115) While stirring the samples on a magnetic stir plate remove subsamples from each of the graduated cylinders and analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C and 50°C from 0 to 300 s^{-1} in 2 minutes and then returning from 300 to 0s^{-1} in 2 minutes. Print out a copy of the rheograms and attach to this test plan.

25°C / 50°C

C6 A File name 062200A Date analyzed 5 wt% 062300A
C6 A Duplicate File name 062200B Date analyzed 062300B 062300C
C6 B File name 062200C Date analyzed 15 wt% 062300D
C6 B Duplicate File name 062200D 062200E Date analyzed 062300E 062300F
C6 C File name 062200F 062200G Date analyzed 25 wt% 062300G*
C6 C Duplicate File name 062200H Date analyzed H I J* Good

116) Return this material to the respective graduated cylinders.

not enough material in H + I

117) Assemble a mixing vessel using the following parts or equivalent. Attach impeller to a stirring motor capable of maintaining a constant rotational rate from 100-1400 rpm.

Part	Vendor	Catalog number
500 ml O-ring Sealed Kettle, 3.75 inch OD, 5 3/8 inch flange	Labglass	LG-8071-100
Clamp	Labglass	LG-7316-106
O-ring	Labglass	LG-1022-476
Kettle top with three 24/40 necks	Labglass	LG-8072-100
2 3/8 diameter 4 blade impeller	Fisher Scientific	14-505-20G

used 2 neck round bottom 250ml - tare 168.78g
stopper A - 14.44g
B - 14.39g
total 197.61g

Can to hold round Bottom 67.76g

Test Plan
29953-073
Back of page 58

Target for slurry mass
in round Bottom = 68.2 g from
page 34
step 72

mass of round Bottom 168.78g
Mass of stoppers ① 14.44 g
② 14.39 g

Total
target → 265.81g

mass @ 8AM on 7/20/00
271.39g → 5.58g too high

after decanting
small amount of
standing liquid @ 8:15 → 266.68g → 0.87 too high
7/20/00

after SrTrn → 284.54 g
addition

net addition = 17.86 g
original material = 20.48 g
lost = 2.6283 g H_2O − 0.87

= target H_2O to add = 1.75
New target = 286.293

after H_2O addition = 286.34g

1:35 Start glass former addition –
1 bottle black
Had to bump power/material is not mixing we
glass formers are floating on top

1:43 – Second bottle of G.F. added – (Video on)
Bumped power to mix

1:52 Start 1hr Stir time ~400rpm

53-6735-200(3-72)

attachment to
29953-073
step 123 p.59

Transfer of C104-25 sample from mixing vessel to aging vessel

7/28/00 Orig. wt. @ Start of mixing = 286.34 g

wt @ end of testing = 247.21 g
w/corks & roundbottom.

Material used for Rheology tests = 39.13 g

Equipment tare (corks & ~~flask~~ flask) = 197.61

$\boxed{\therefore\ 49.6\ g}$ material left in flask assuming no evaporation loss

Tare of New sample bottle - 134.11 g

Removed all the material possible without ~~xxx~~ extra H_2O new wt = 156.52 g
= 22.41 g sludge

Not Enough have to rinse and evaporate

after rinsing equipment tare = 198.64 g
Start tare = 197.61 g
= 1.03 g material lost

calculated
→ Material left in flask = 49.6 g
− 1.03
48.57 g wanted Material in jar C-104-25-2-final
+ tare = 134.11 g
= 182.68 g = target wt of jar

7/28/00 3:25 PM
actual wt. 279.66 g

Need to evaporate 96.98 g H_2O

• mixing study conducted on C104-25-2 ...
• mass on 7/11/00 = 197.307g

118) Transfer the sample specified by BNFL to the mixing vessel (do not transfer the stir bar). Record which sample was transferred as well as the day and time transferred below.

Sample transferred 7/18/00 Date C104-25-2 Time Am

119) Turn on the stirrer and adjust the rotational speed to that specified by BNFL. Record the time, date and speed below.

Speed ~400 rpm rpm Date 7/20/00 Time 1:52 pm

120) After 1 hour of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to 300 s^{-1}. Print out a copy of the rheograms and attach to this test plan. re ran standard 95.5cP using MUF @ on 7/20/00 File 072000A

35→25 C - temp change potential

Run 1 File name 072000B Date analyzed 7/20/00 ~3:25 pm

Run 2 File name 072000C Date analyzed 7/20/00 ~3:30 pm sample little overfilled

072000D 7/20/00 ~3:35 FE 3:45 restarted FE 4:00

121) After 1 day of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to 300 s^{-1}. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name 072100A Date analyzed 7/21/00 ~3:20

Run 2 File name 072100B Date analyzed 7/21/00 ~3:30

072100C / 072100D ~3:35 & 3:40

122) After 1 week of stirring, remove a sample through the sampling port and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to 300 s^{-1}. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name 072700A Date analyzed 7/27/00 ~

Run 2 File name 072700B Date analyzed 7/27/00 ~

123) After removing the last sample (after 1 week of stirring), transfer the sample back to the graduated cylinder. Record sample ID, volume and mass of material.

C-104-25-2 - Final tare = 134.11g

C6 ___ mass on 8/3/00

Total	182.68	g	Total	~~___~~	ml
Tare	134.11	g	Solids	~~___~~	ml
Slurry	48.57	g	Liquids	~~___~~	ml

Could not read accurately due to dirty walls

124) Focus a video camera on the solids-liquid interface of the sample and collect video for one week taking care not to disturb the sample. Report any observed gas retention and/or release behavior to the cognizant scientist.

125) After the sample has remained undisturbed for 1 week, remove the standing liquid using a glass

No bubbles seen releasing or trapped in sludge

Tare C104-25-2 wash 129.07 g

pipette.

126) Gently collect a subsample of the settled solids and immediately analyze for shear stress as a function of shear rate in duplicate. Conduct the analysis at 25°C from 0 to 300 s^{-1}. Print out a copy of the rheograms and attach to this test plan.

Run 1 File name 081000A Date analyzed 8/10/00

Run 2 File name 081000B Date analyzed 8/10/00

081000C 8/10/00

PNNL Test Plan Addendum 1

Document No.: BNFL-TP-29953-073 Add 1
Rev. No.: 0
Document Control: Only the original signed copy is controlled

Title: LAW Melter Feed Rheological and Physical Properties Measurements

Work Location: Radiochemical Processing Laboratory

Author: Paul Bredt

Effective Date: Upon Final Approval
Supersedes Date: New

Use Category Identification: Reference

Identified Hazards:
- __ Radiological
- __ Hazardous Materials
- __ Physical Hazards
- __ Hazardous Environment
- __ Other:

Required Reviewers:
- X Technical Reviewer
- X Project Manager
- __ Building Manager
- __ RPL Manager
- __ Radiological Control
- __ SFO Manager
- __ ES&H
- __ Quality Engineer

Are One-Time Modifications Allowed to this Procedure? X Yes __ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? _____ Yes or X No

FOR REVISIONS:
Is retraining to this procedure required? _____Yes X No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
_____Yes _____No X N/A

Approval	Signature	Date
Author	Paul Bredt	5/1/00
Technical Reviewer	Harry D. Smith	5/5/00
Project Manager	Eugene V. Morrey	5/11/00
BNFL	[signature]	5/16/00

High Activity Melter Feed Rheological and Physical Properties Measurements

Instructions

Under Test Plan BNFL-29953-073, "High Activity Melter Feed Rheological and Physical Properties Measurements", samples of the C-104 and AZ-102 melter feeds were adjusted to 5, 15, and 25 wt% solids. Duratek has provided BNFL with specifications for secondary waste product and glass former addition to the initial feeds (20 wt% for C-104 and 9.54 wt% for AZ-102). This addendum to the test plan defines specifications for secondary waste product and glass formers additions to the adjusted concentrations.

Secondary waste products to be added to the C-104 waste include Cs eluant and Sr/TRU precipitate. Given the limited quantity of actual secondary waste products currently available, simulated secondary waste products will be used for C-104 testing. Secondary waste products to be added to AZ-102 include Cs eluant, Tc eluant, and Sr/TRU precipitate. Actual secondary waste products will be used for AZ-102 testing.

The Cs eluant simulant will be prepared using the Concentrated Cs Eluant data provided on page 25 of the SRTC report "Estimation of Physical Properties of Tank 241-AN-107 Cesium and Technetium Eluate Concentrate Blend" by Alexander S. Choi. This Cs eluant is approximately 0.14 g of oxide/ml of simulant and 27 wt% dry solids.

The Sr/TRU simulant will be prepared from an AN-107 simulant. The Sr/TRU simulant will be adjusted to 25 wt% solids prior to addition. Both the Sr/TRU precipitate and Cs eluant will be analyzed by ICP prior to addition to the actual waste.

Table 1 provides the specifications for a 400 g sample of C-104 at 20 wt% solids. This is the basis used in Test Instruction 29953-079 Rev.1 that reflects the Duratek specification. Table 1 also provides ratios required for addition to the adjusted samples. The 400 g basis will be adjusted to the actual sample masses at the time of additions.

Table 2 provides the specifications for a 436.27 g sample of AZ-102 at 9.535 wt% solids. This is the basis used in Test Instruction 29953-079 Rev. 1 Addendum 5 that reflects the Duratek specification. Since the AZ-102 sample masses are known, Table 2 defines exactly how much of each material is to be added to each of the samples.

Table 3 lists the AZ-102 samples received for this testing. At the conclusion of the AZ-102 testing, all AZ-102 material will be composited into a single jar labeled "**AZ-102 Melt 2 COMP**" for transfer to the vitrification task.

The quantity of Cs eluant and Tc eluant required for the AZ-102 process baseline is very small. The Cs and Tc eluants are to be added at 27 and 25 wt% solids respectively, and they are currently less than approximately 0.2 and 0.02 wt% solids respectively. Therefore, following evaporation to the correct water content, these small additions will not effect the rheological properties of the test materials.

For example, the largest addition would be less than 0.32g of Cs eluant addition to the 136.56 g sample Rheol-25-1:

(137.83 g eluant/436.69g waste) x (0.002 g solids/g eluant) x (1 g concentrated eluant /0.27 g solids) x (136.56 g waste) = 0.319 g concentrated eluant

Given the small amounts of concentrated eluants required, the Cs and Tc eluants will not be added for rheological testing. The diluted eluants (137.83 and 76.67 g respectively) will be added directly to the final sample "**AZ-102 Melter 2 COMP**" prior vitrification task.

Table 1. Secondary waste products and glass formers to be added to C-104 melter feed.

Additive	Target (g) per 400g of Slurry			
	20 wt%	5 wt%	15 wt%	25 wt%
Sr/TRU Precipitate (dry solids basis)	25.674	6.418	19.256	32.092
Cs Eluant (27 g total solids/ml solution)	8.8 ml	2.2 ml	6.6 ml	11 ml
$Na_2B_4O_7+10H_2O$	58.000	14.500	43.500	72.500
$LiOH+H_2O$	36.800	9.200	27.600	46.000
Na_2CO_3	3.525	0.88125	2.64375	4.406
SiO_2 (Sil-co-Sil 75)	116.280	29.070	87.210	145.350
ZnO (KADOX-920)	4.93	1.232	3.698	6.162
Sugar (Granular)	0	0	0	0

Table 2. Secondary waste products and glass formers to be added to AZ-102 Melt-2 melter feed in grams. The AZ-102 Slurry Basis is 9.535 wt% solids.

Sample	Slurry Basis	Rheol-5	Rheol-15	Rheol-25-1	Rheol-25-2
Slurry Mass (g)	436.69	41.84	122.00	136.53	136.32
Sr/TRU Precipitate (25 wt% solids basis)	10.38g x (46.26 wt%/25 wt%) = 19.21	1.841	5.367	6.006	5.997
Actual Cs Eluant (Diluted Basis)	137.83	13.206	38.506	43.092	43.026
Actual Tc Eluant (Diluted Basis)	76.67	7.346	21.420	23.971	23.934
$Na_2B_4O_7+10H_2O$	10.76	1.031	3.006	3.364	3.359
$LiOH+H_2O$	15.51	1.486	4.333	4.849	4.842
$Na_2SiO_3+5H_2O$	32.09	3.075	8.965	10.033	10.017
SiO_2 (Sil-co-Sil 75)	39.73	3.807	11.100	12.421	12.402
Sugar (Granular)	1.34	0.128	0.374	0.419	0.418
		9.527 ✓	27.778 ✓		

PRB 5/24/00

Table 3. Samples of AZ-102 slurry received for testing. Samples are 9.535 wt% solids.

Sample	Mass (g)
AZ-102 Washed Sludge:Rheol-5	41.84
AZ-102 Washed Sludge:Rheol-15	122.00
AZ-102 Washed Sludge:Rheol-25-1	136.53
AZ-102 Washed Sludge:Rheol-25-2	136.32

PNNL Test Instruction To Supplement TP-29953-073	**Document No.: BNFL-TP-29953-073** **Rev. No.: 0** **Document Control: Only the original signed copy is controlled**
Title: High Activity Melter Feed Rheological and Physical Properties Measurements	
Work Location: Radiochemical Processing Laboratory	**Page** 1 of 3
Author: Paul Bredt	**Effective Date:** Upon Final Approval **Supersedes Date: New**
Use Category Identification: Reference	
Identified Hazards: __ Radiological __ Hazardous Materials __ Physical Hazards __ Hazardous Environment __ Other:	**Required Reviewers:** X Technical Reviewer __ Building Manager __ Radiological Control __ ES&H __ Quality Engineer __ Project Manager __ RPL Manager __ SFO Manager

Are One-Time Modifications Allowed to this Procedure? X Yes __ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? _____ Yes or X No

FOR REVISIONS:
Is retraining to this procedure required? _____Yes X No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
_____Yes _____No X N/A

Approval	**Signature**	**Date**
Author	Paul Bredt	6/13/00
Technical Reviewer	Harry A. Smith	6/13/00

High Activity Melter Feed Rheological and Physical Properties Measurements

Scope

This test instruction provide specific details regarding the implementation of Test Plan BNFL-29953-073 Addendum 1, "High Activity Melter Feed Rheological and Physical Properties Measurements". Addendum 1 to this Test Plan, provides the ratio of glass formers, and secondary waste products to be added to each of the AZ-102 melter feed samples. However, due to changes in client needs, not all of the steps in the Test Plan were implemented. As a result, the samples do not contain all ingredient required for the sample transfer to the Vitrification Task as specified in Test Plan 29953-073 Addendum 1. This test Instruction provides the details required to prepare the feed for the Vitrification Task.

Work Instructions

1) Weigh a clean ~~500 ml~~ glass jar labeled **AZ-102 Melter 2 COMP.**
1 liter PRB 6/14/00

AZ-102 Melter 2 COMP

Tare 468.71 g ← without metal band and without tape on lid
with tape on lid w/out Band → 469.46 g

2) Transfer the material in **AZ-102 Rheol-5, AZ-102 Rheol-15,** and **AZ-102 Rheol-25-1** to **AZ-102 Melter 2 COMP.** Use deionized water as needed to transfer the material.
with band & tape 528.32 g

3) Weigh 25.116g of AZ-102 Glass formers from the jar labeled **AZ-102 Glass** to a clean 40 ml vial or similar container. Weigh the material in the vial.

	Clean Vial	
Total	49.6894	g
Tare	24.5595	g
Solids	25.1299	g

using 384-06-01-004 exp. 8/2000

4) Transfer the glass formers collected in the previous step to **AZ-102 Melter 2 COMP.** added
6/14/00

5) Weigh 2.33g of material from the jar labeled **AN-107 DF SOLIDS** to a clean 20 ml vial or similar container. Weigh the material in the vial.

used tared plastic boat

	Clean Vial	
Total		g
Tare		g
Solids	~~2.364~~ 2.367 PRB 6/15/00	g

using 360-06-01-054 exp 8/00 for rest of Test Instruction.
PRB 6/14/00

6) Transfer the **AN-107 DF SOLIDS** collected in the previous step to **AZ-102 Melter 2 COMP.**

7) Weigh 86.5g of Cs eluant into a clean 125 ml jar or similar container. Weigh the material in the jar. Record the label on the original Cs eluant bottle.

Cs Eluant Bottle "Cs 1of2 composite 3/00" mass on 6/14/00 = 733.33g before removing material PRB 6/14/00
Bottle Tare = 355.44g

	Clean Jar
Total	203.477 g
Tare	116.98 g
Solids	86.497 g

8) Transfer the Cs eluant collected in the previous step to **AZ-102 Melter 2 COMP.**

9) Weigh 48.133g of Tc eluant into a clean 125 ml jar or similar container. Weigh the material in the jar. Record the label on the original Cs eluant bottle.

Tc Eluant Bottle "AZ-102 Tc Melt #2" mass on 6/14/00 = 210.485g before removing material PRB 6/14/00
Tare = 134.080g

	Clean Jar
Total	164.900 g
Tare	~~116.98~~ g 116.708
Solids	48.192 g

10) Transfer the Tc eluant collected in the previous step to **AZ-102 Melter 2 COMP.**

11) Mix the material in **AZ-102 Melter 2 COMP** and describe the mixing technique and observations below.

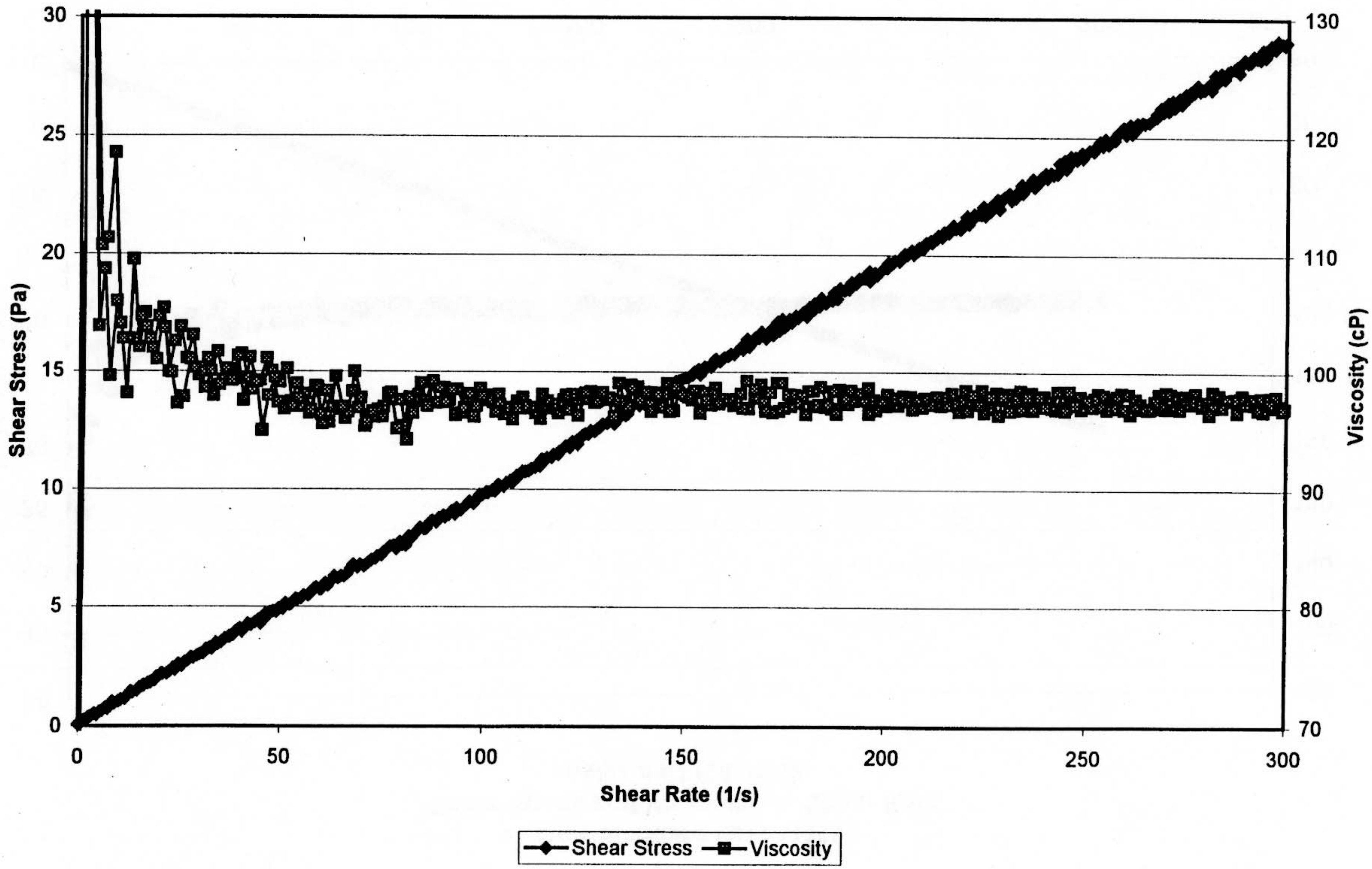
sample mixed using a Braun hand blender modified for hot cell operation for ~1min.
6/15/00
PRB
@ 9:15 am

164.89

APPENDIX B

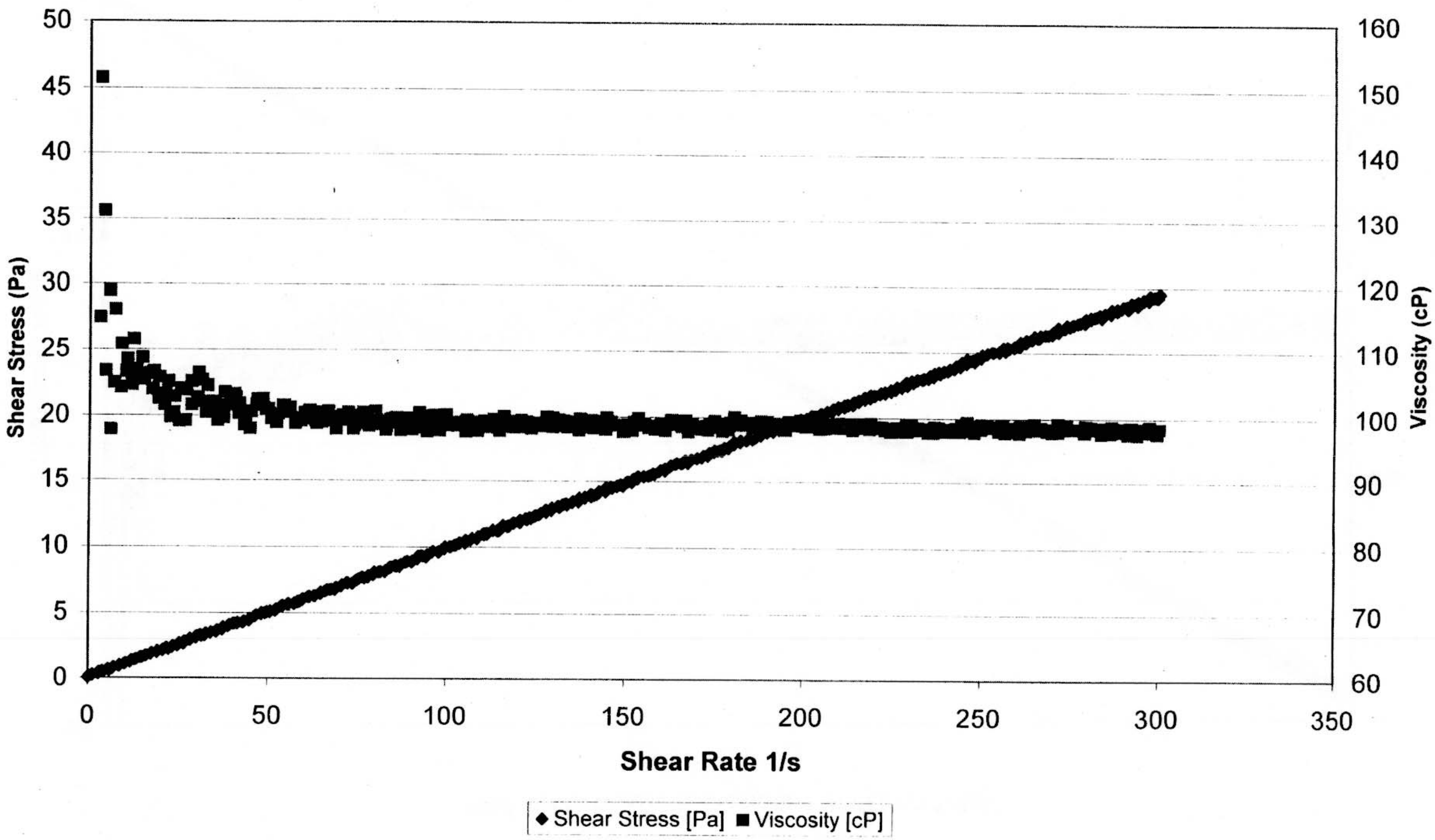
Appendix B: Rheograms

95.5 cP Brookfield Standard: 25°C on 5/2/00

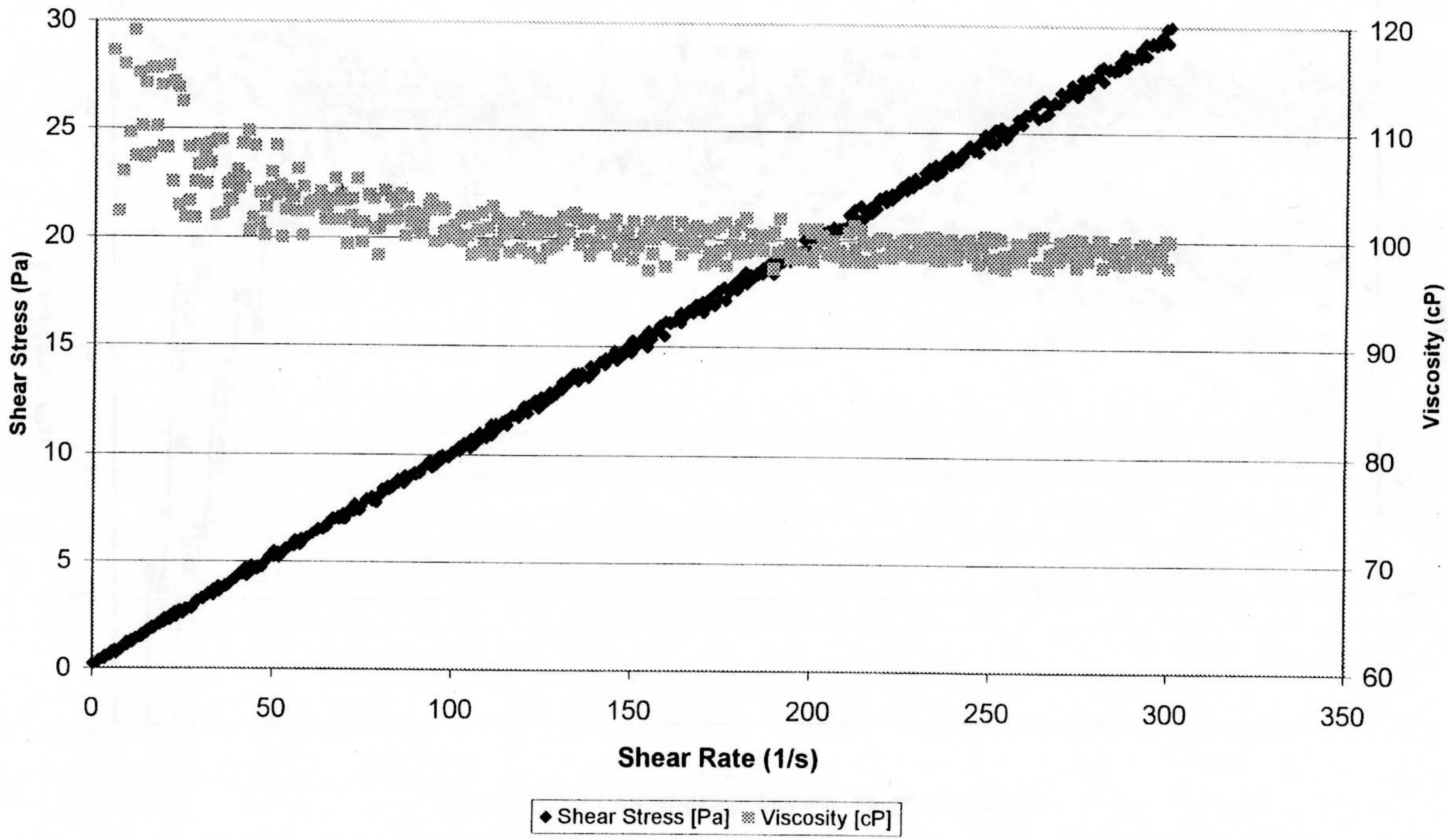


Haake M5 Viscometer with MV1 Geometry

95.5 cP Standard Lot #111199
Shear Stress and Viscosity vs. Shear Rate
Analysis #1 (06/01/00)

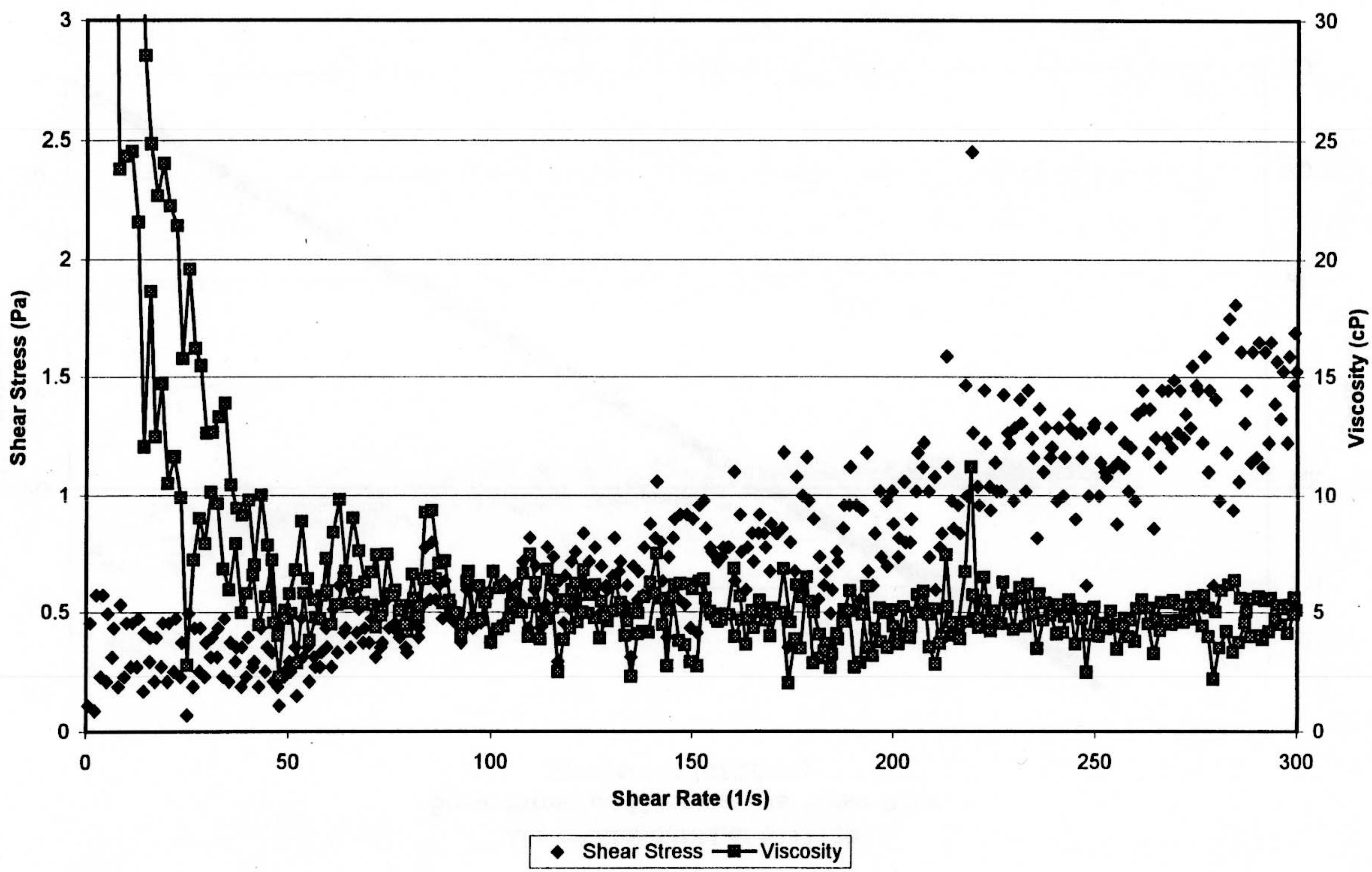


95.5 cP Standard Lot #111199
Shear Stress and Viscosity vs. Shear Rate
Analysis #1 (07/20/00)



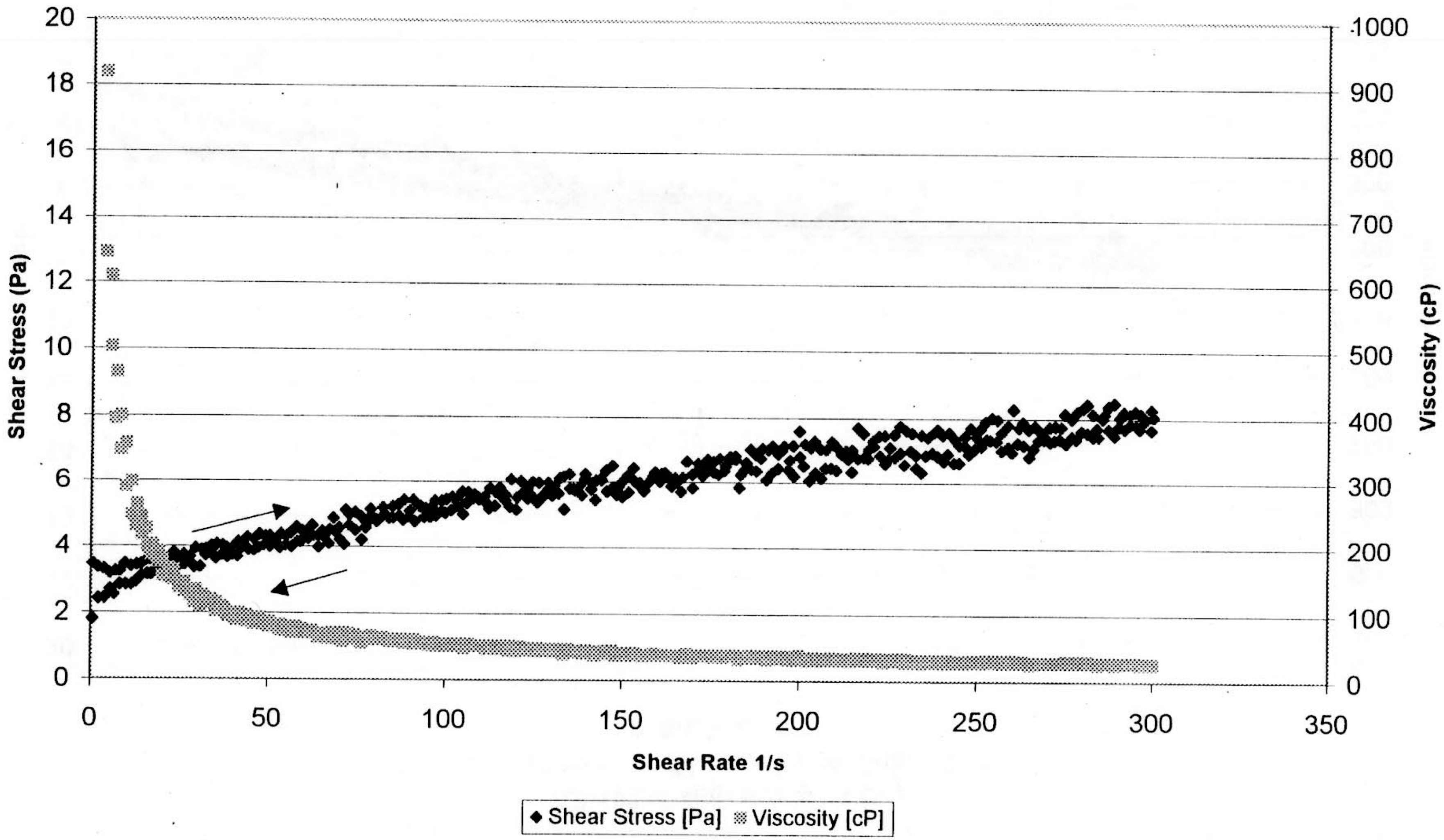
Measurement performed with a Haake M5 and MV I sensor

AZ-102 Melter Feed 5wt% Solids: 25°C, Analysis 1

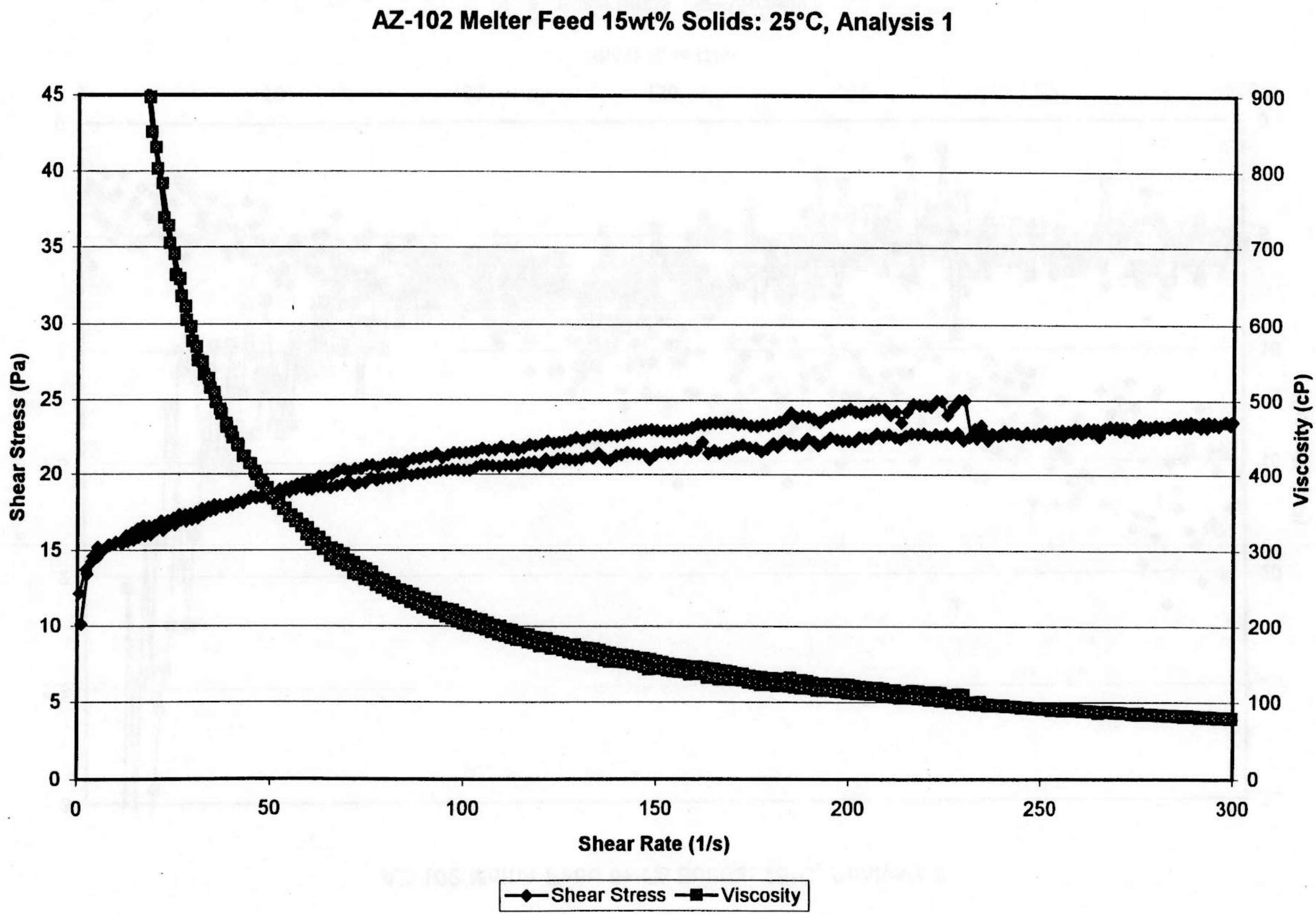


Haake M5 Viscometer with SV1 Geometry

AZ-102 Mixing Study - 1 Day
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

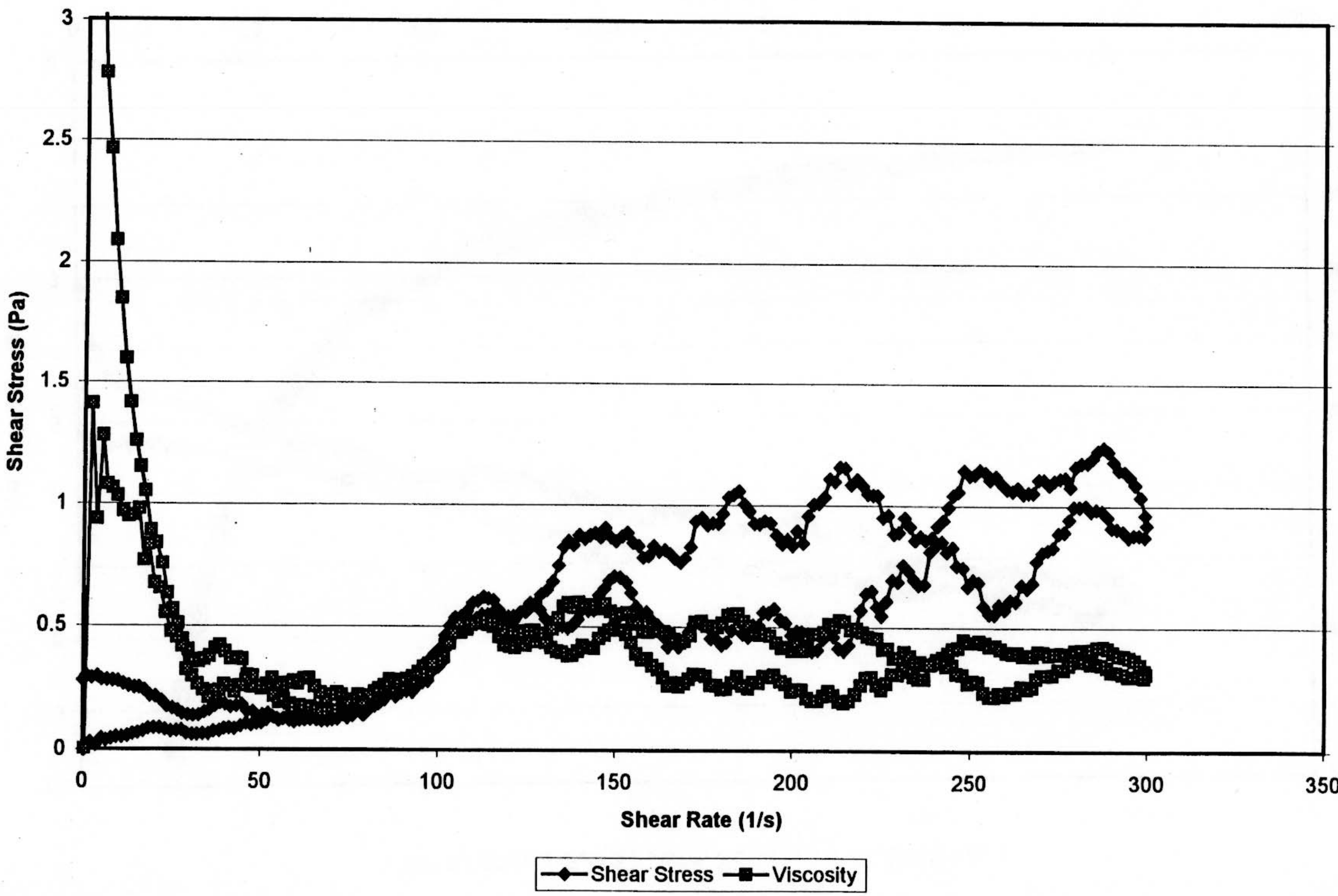


Measurement performed with a Haake M5 and SV I sensor

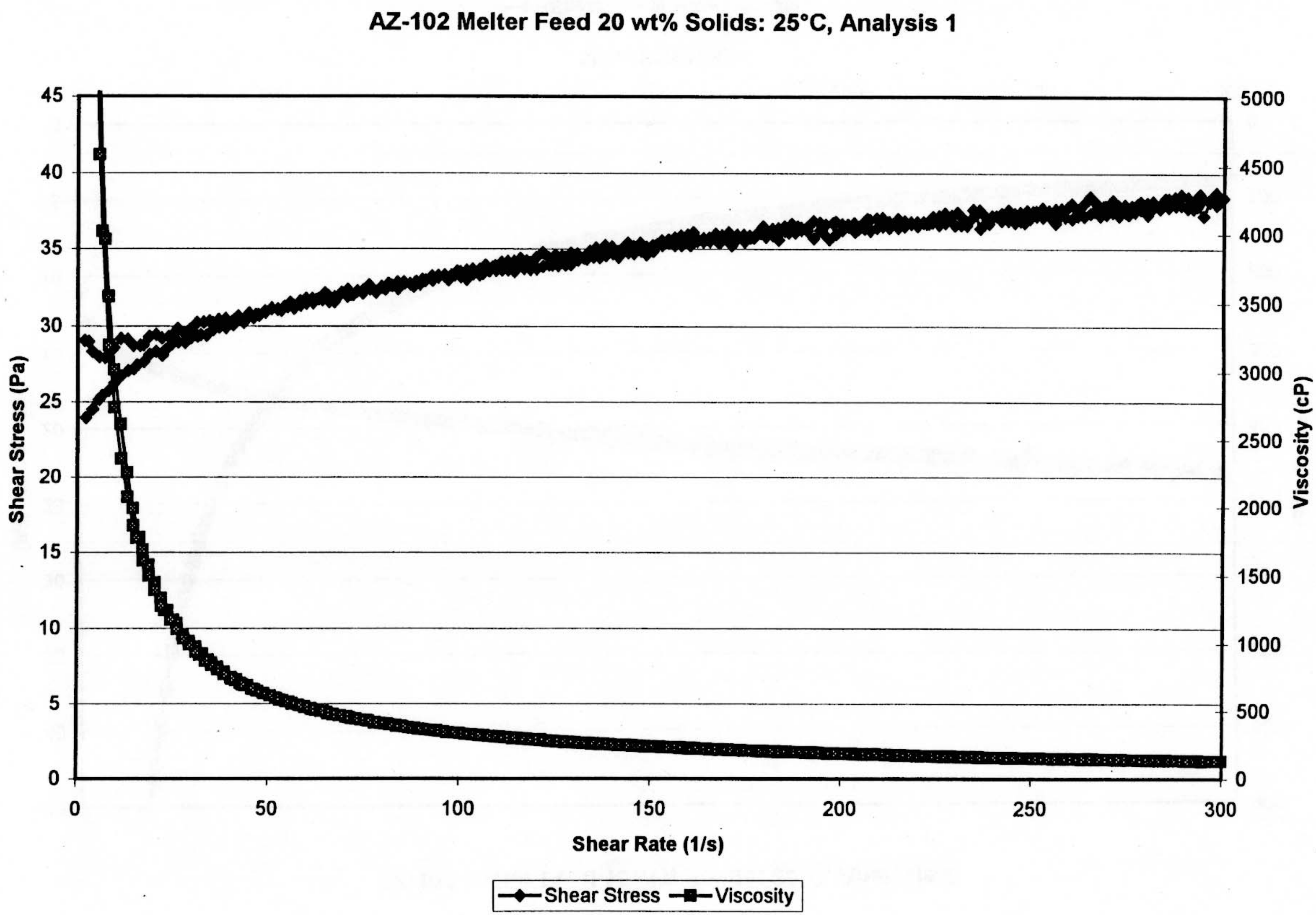


Haake M5 Viscometer with SV1 Geometry

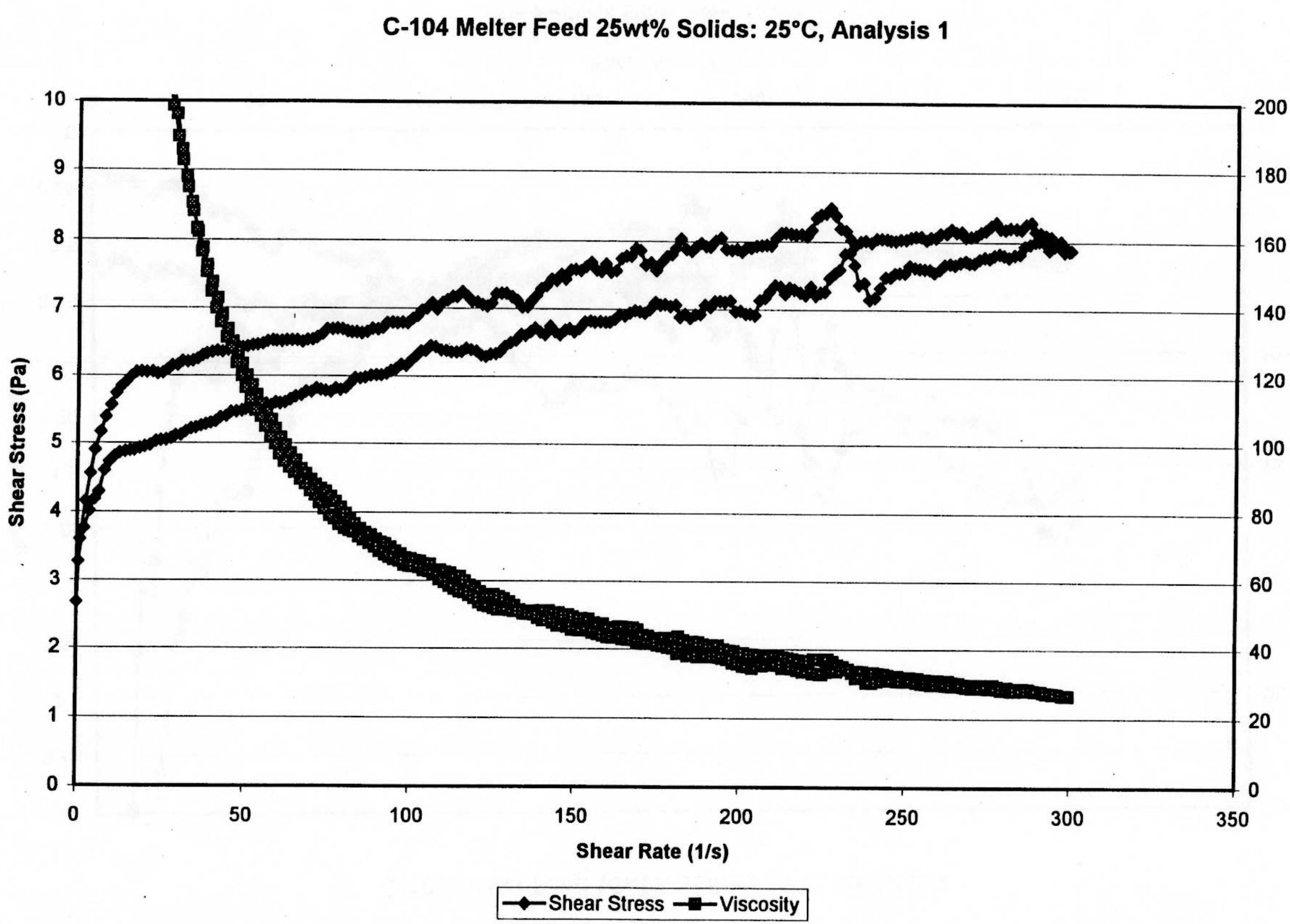
C-104 Melter Feed 5wt% Solids: 50°C, Analysis 1



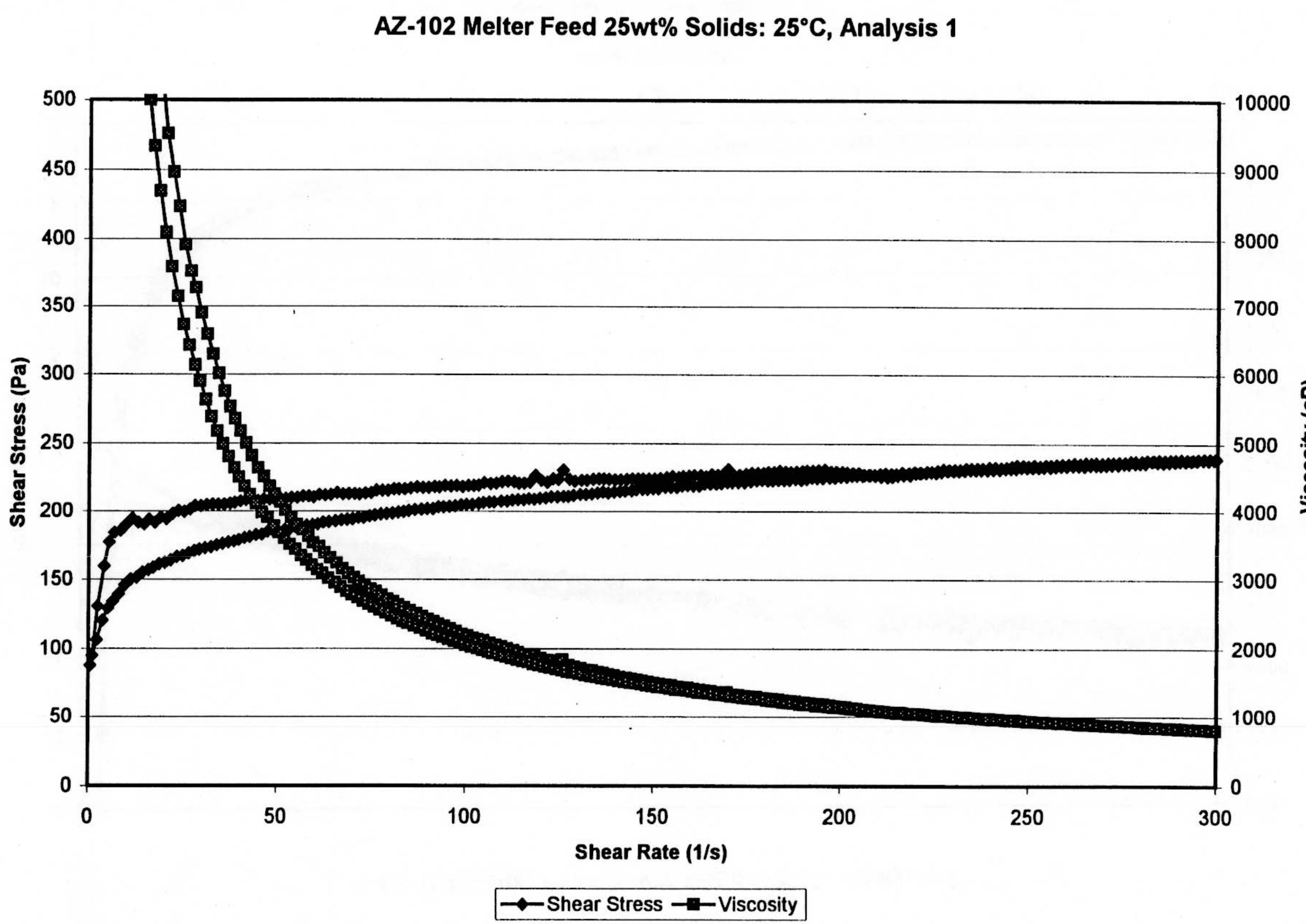
Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry



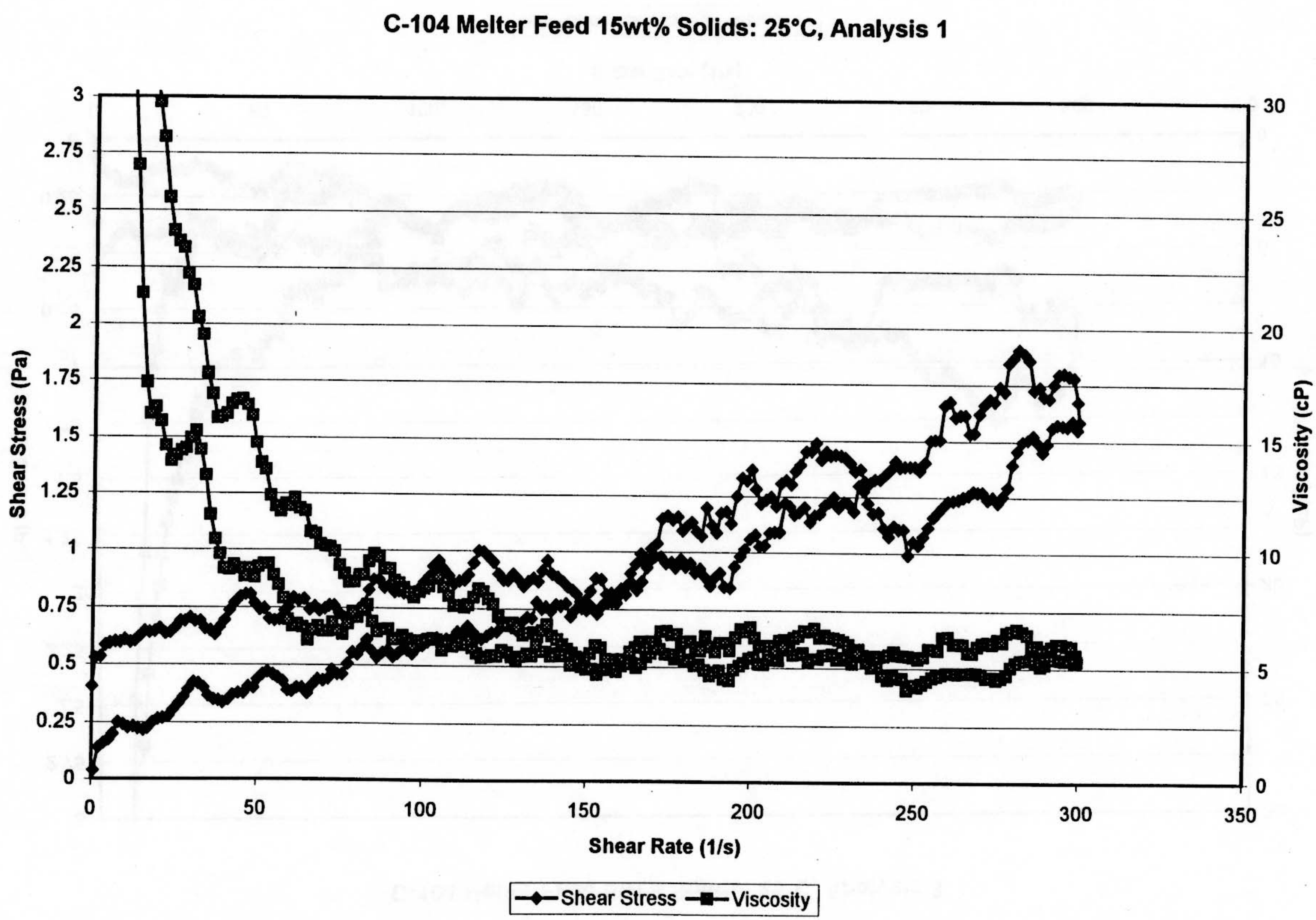
Haake M5 Viscometer with SV1 Geometry



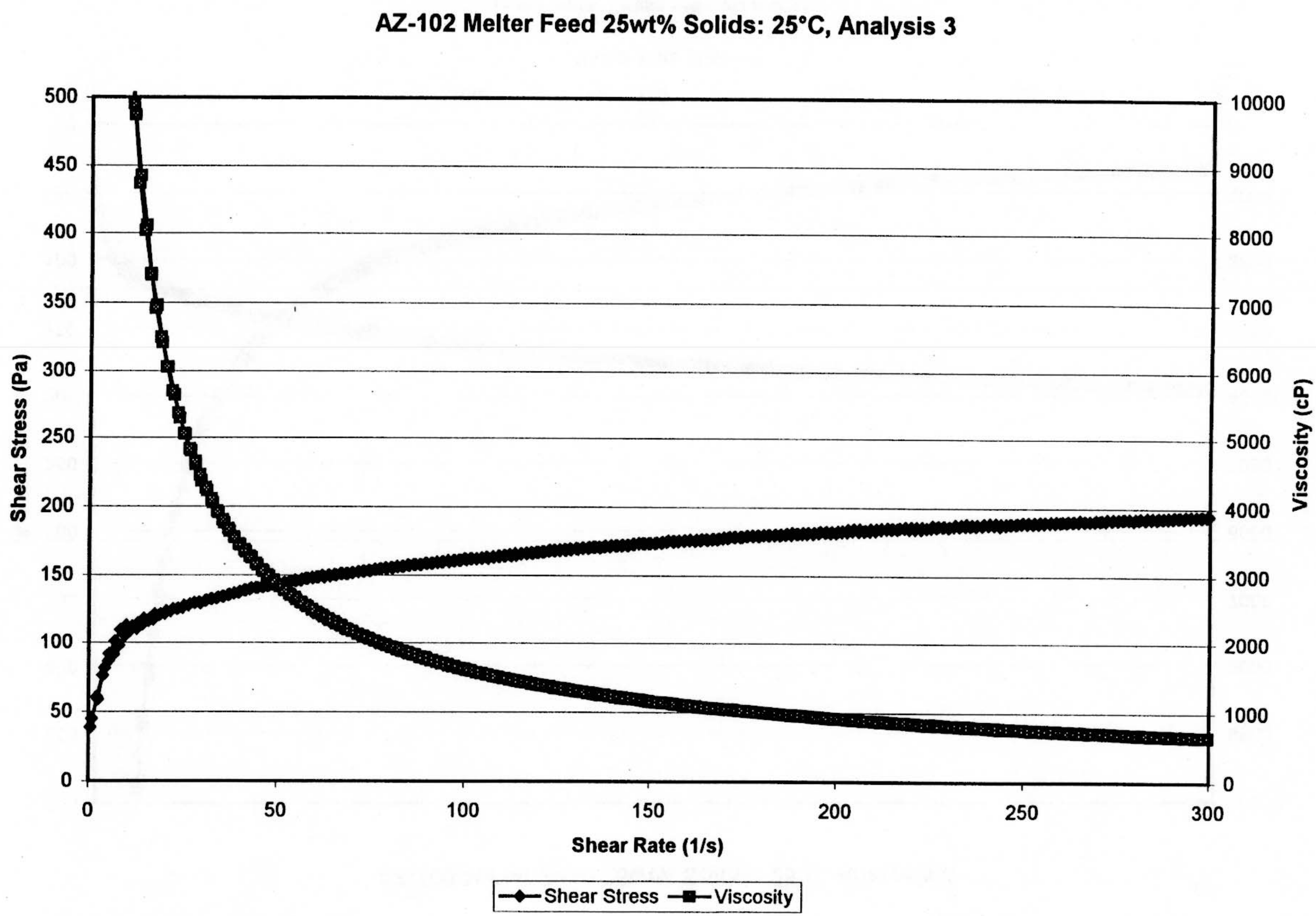
Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry



Haake M5 Viscometer with SV1 Geometry

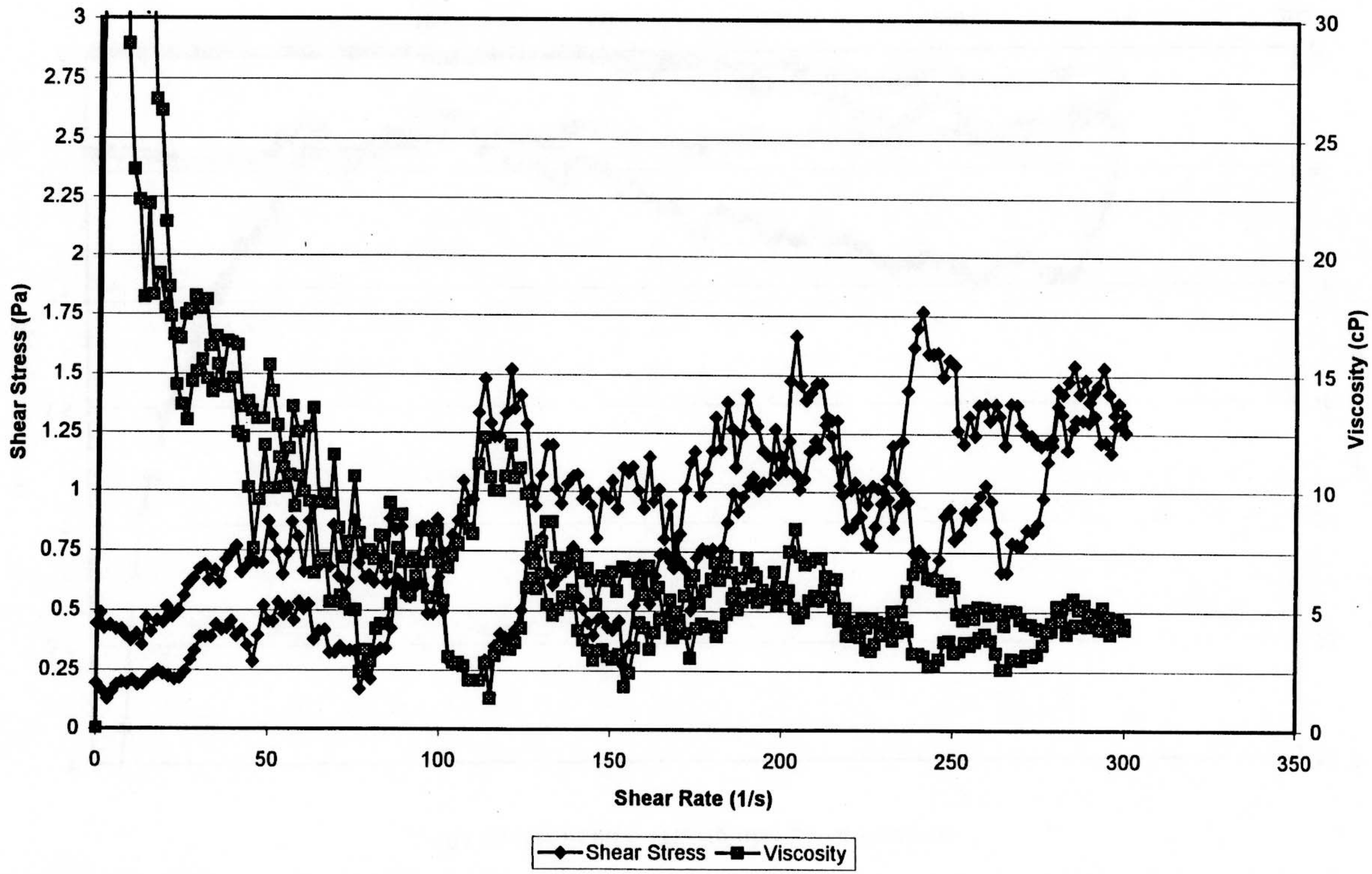


Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry

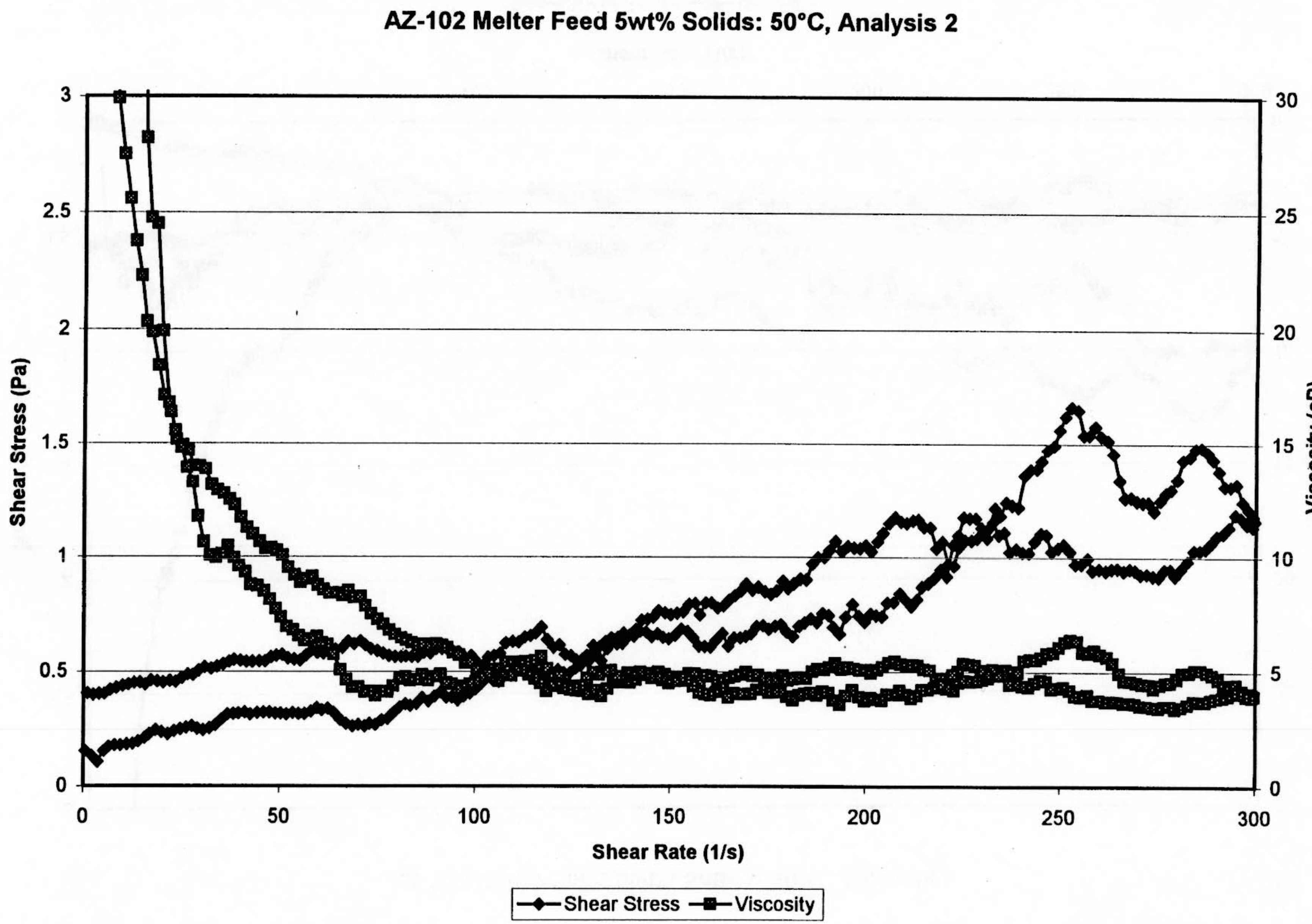


Haake M5 Viscometer with SV1 Geometry

C-104 Melter Feed 5wt% Solids: 25°C, Analysis 2

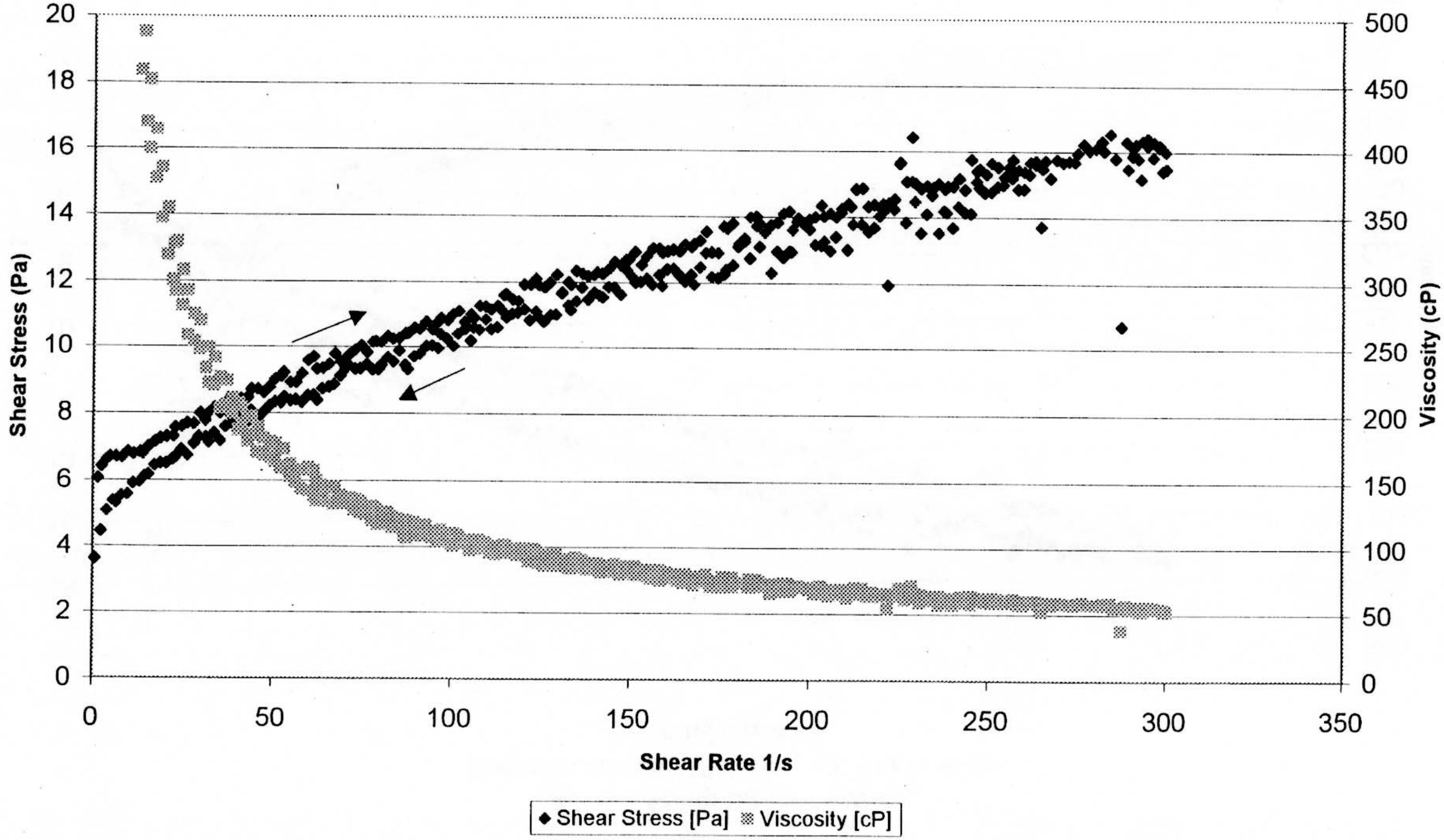


Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry

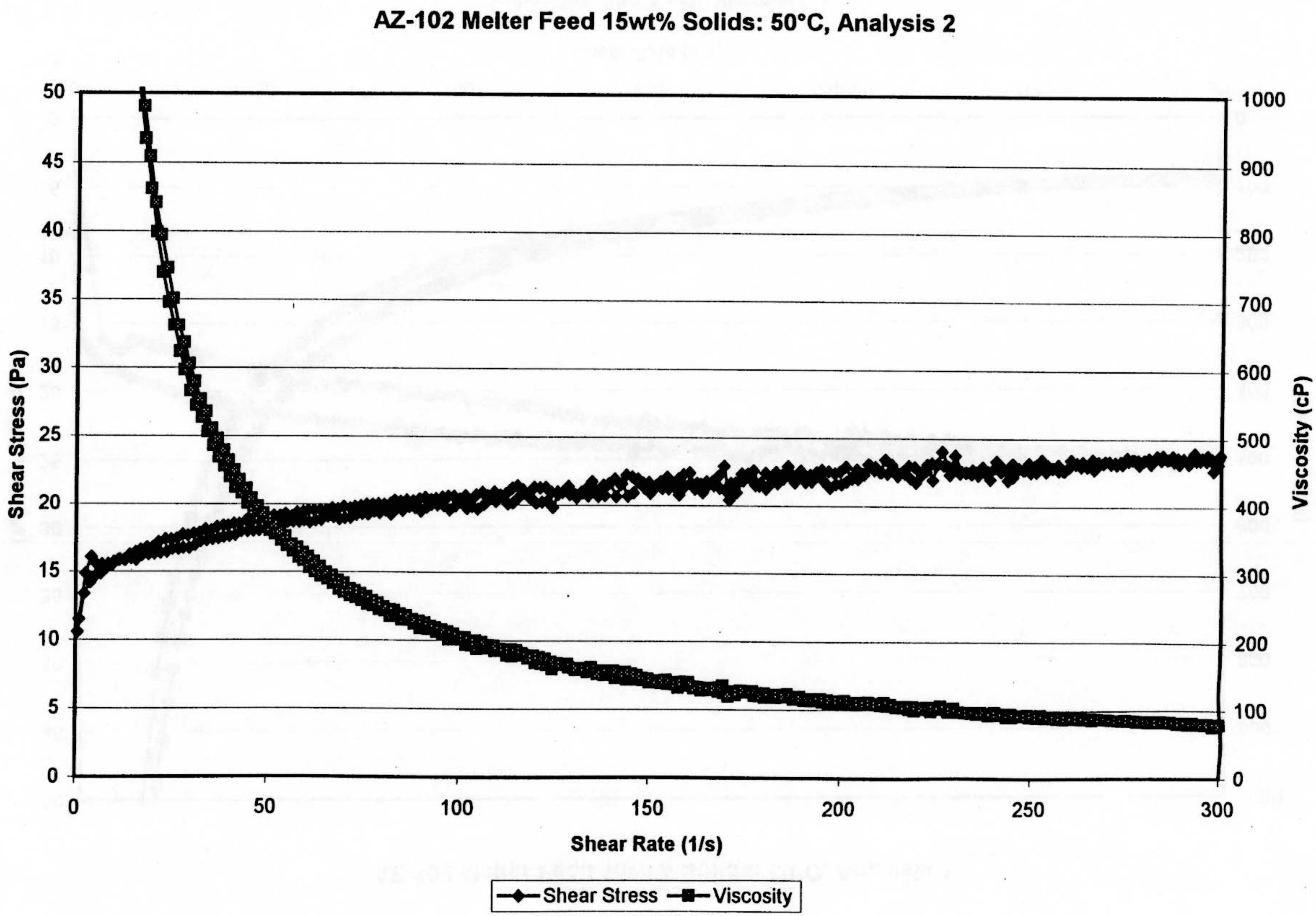


Haake M5 Viscometer with SV1 Geometry

AZ-102 Aging Study - 1 Week
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

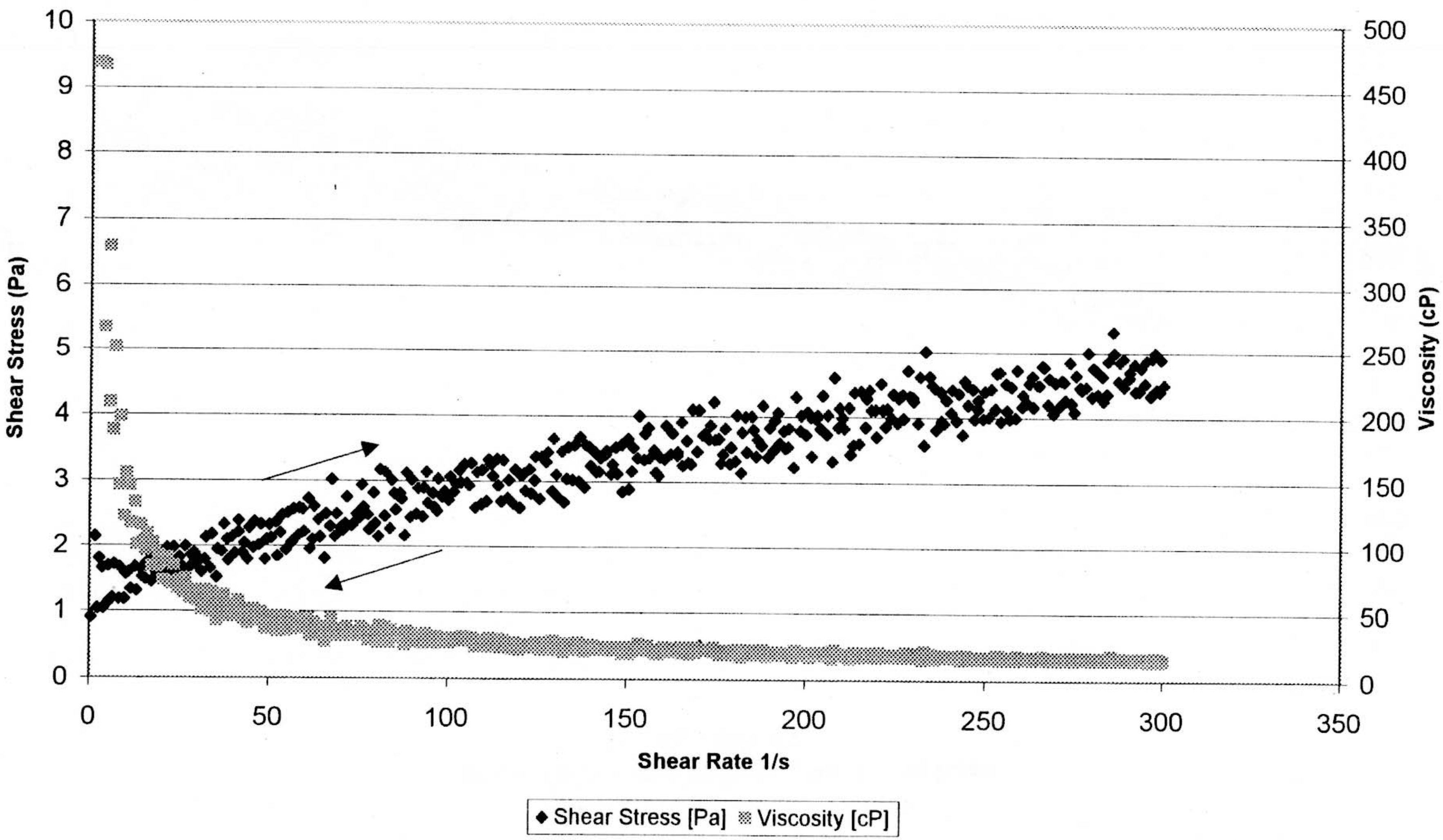


Measurement performed with a Haake M5 and SV I sensor



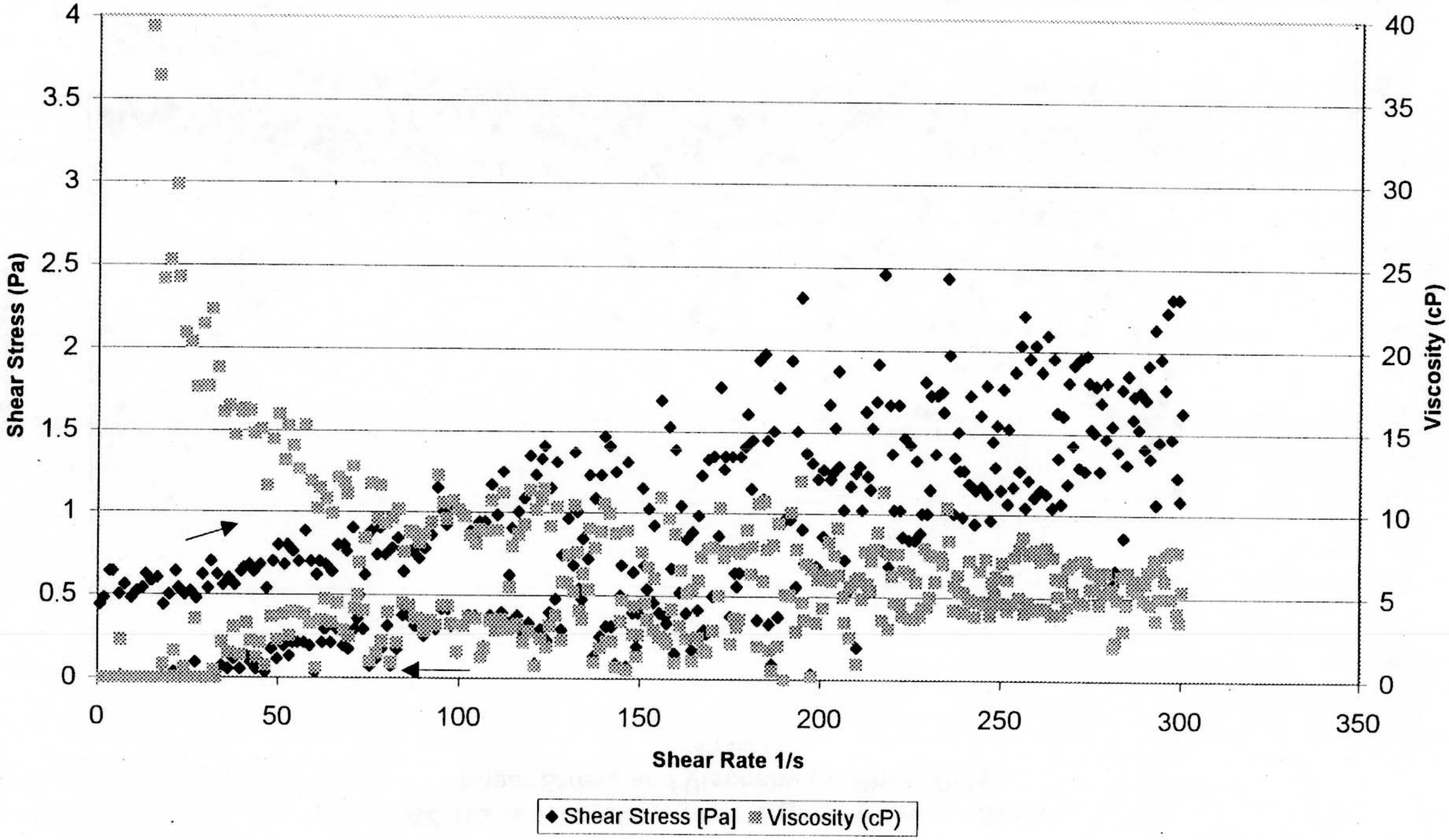
Haake M5 Viscometer with SV1 Geometry

AZ-102 Mixing Study - 1 Week
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



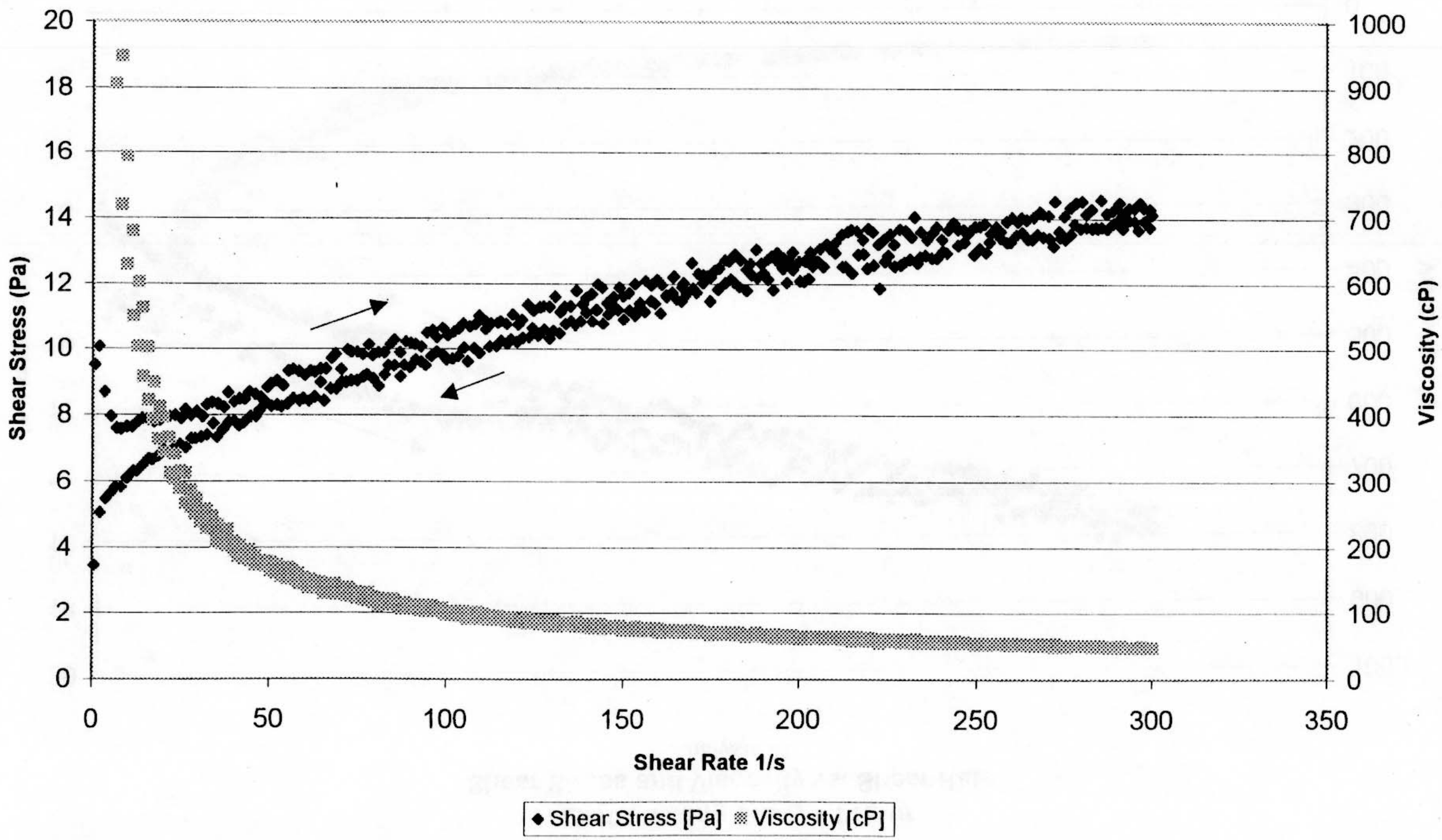
Measurement performed with a Haake M5 and SV I sensor

AZ-102 w/secondary waste & glass formers - 5wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



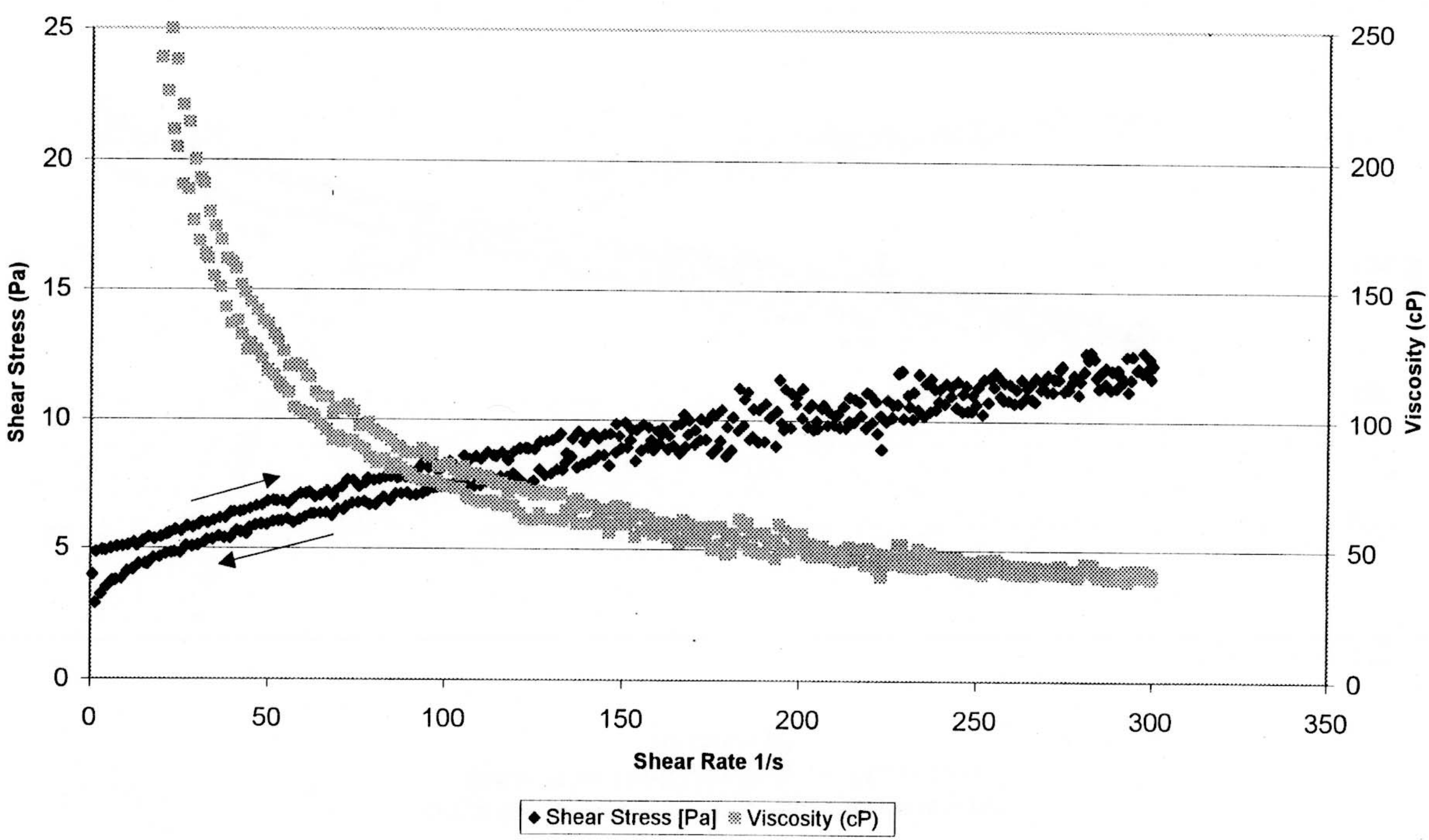
Measurement performed with a Haake M5 and SV I sensor

AZ-102 Mixing Study - 1 Hour
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

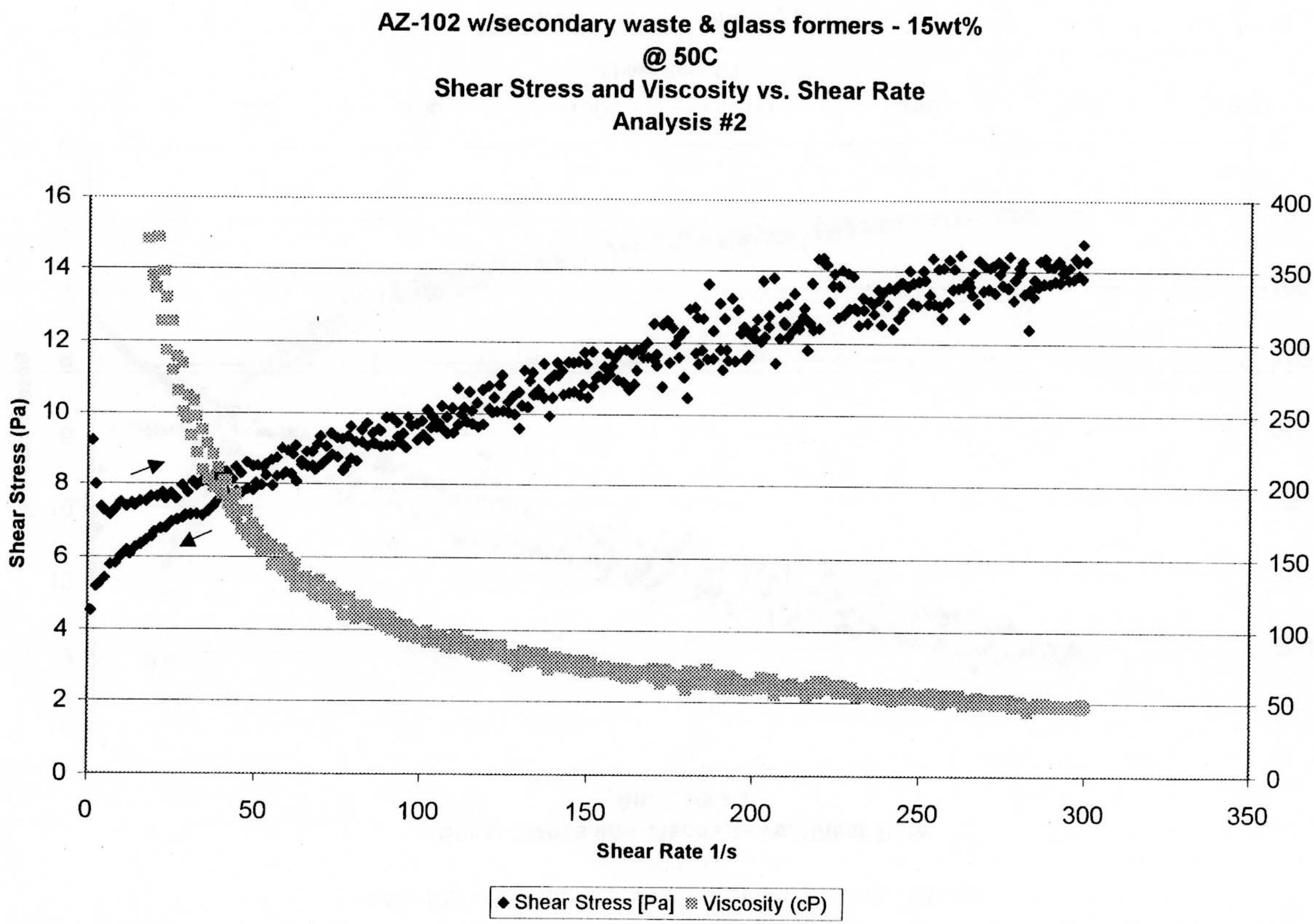


Measurement performed with a Haake M5 and SV I sensor

AZ-102 w/secondary waste & glass formers - 15wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

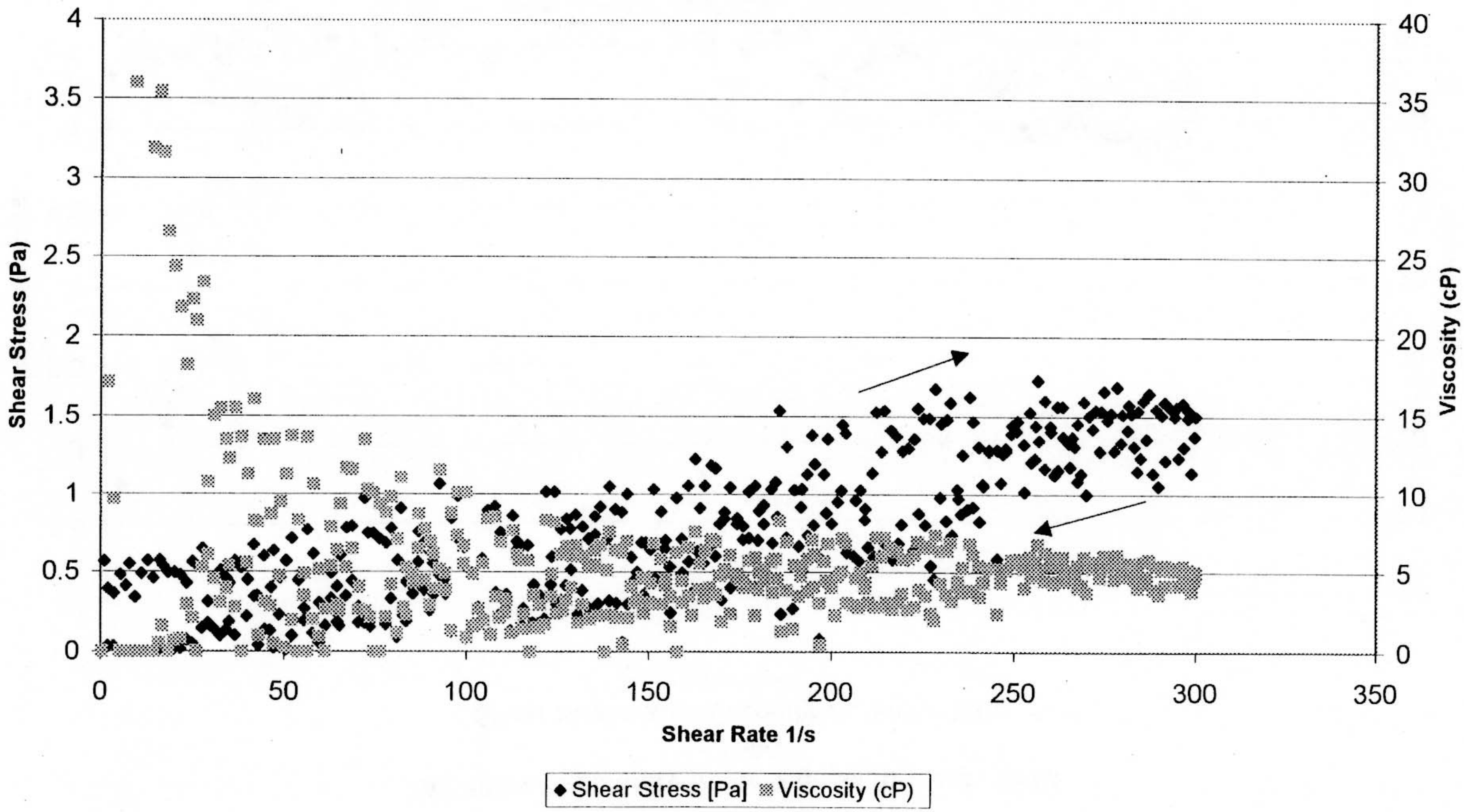


Measurement performed with a Haake M5 and SV I sensor



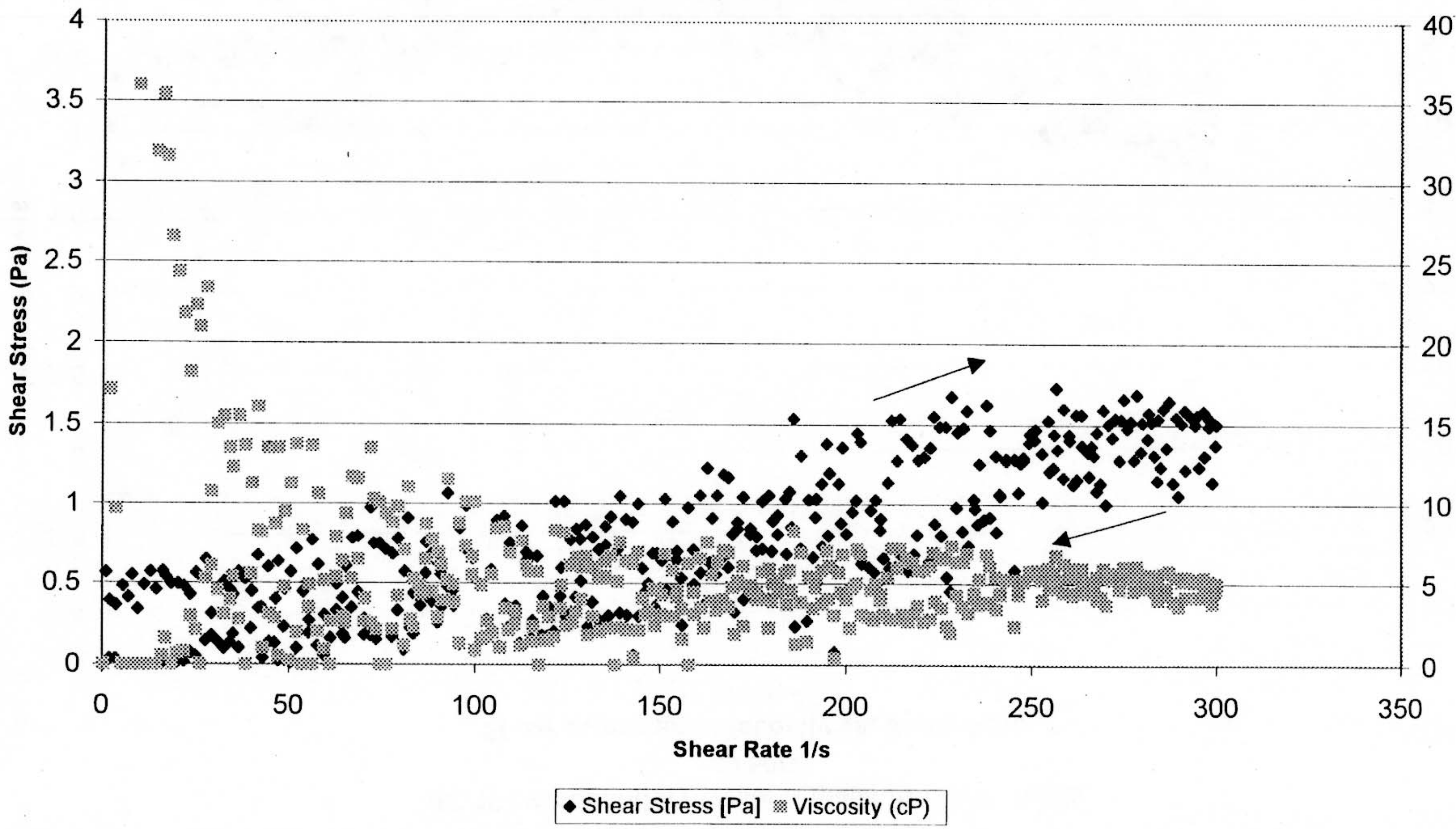
Measurement performed with a Haake M5 and SV I sensor

AZ-102 w/secondary waste & glass formers - 5wt%
@ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



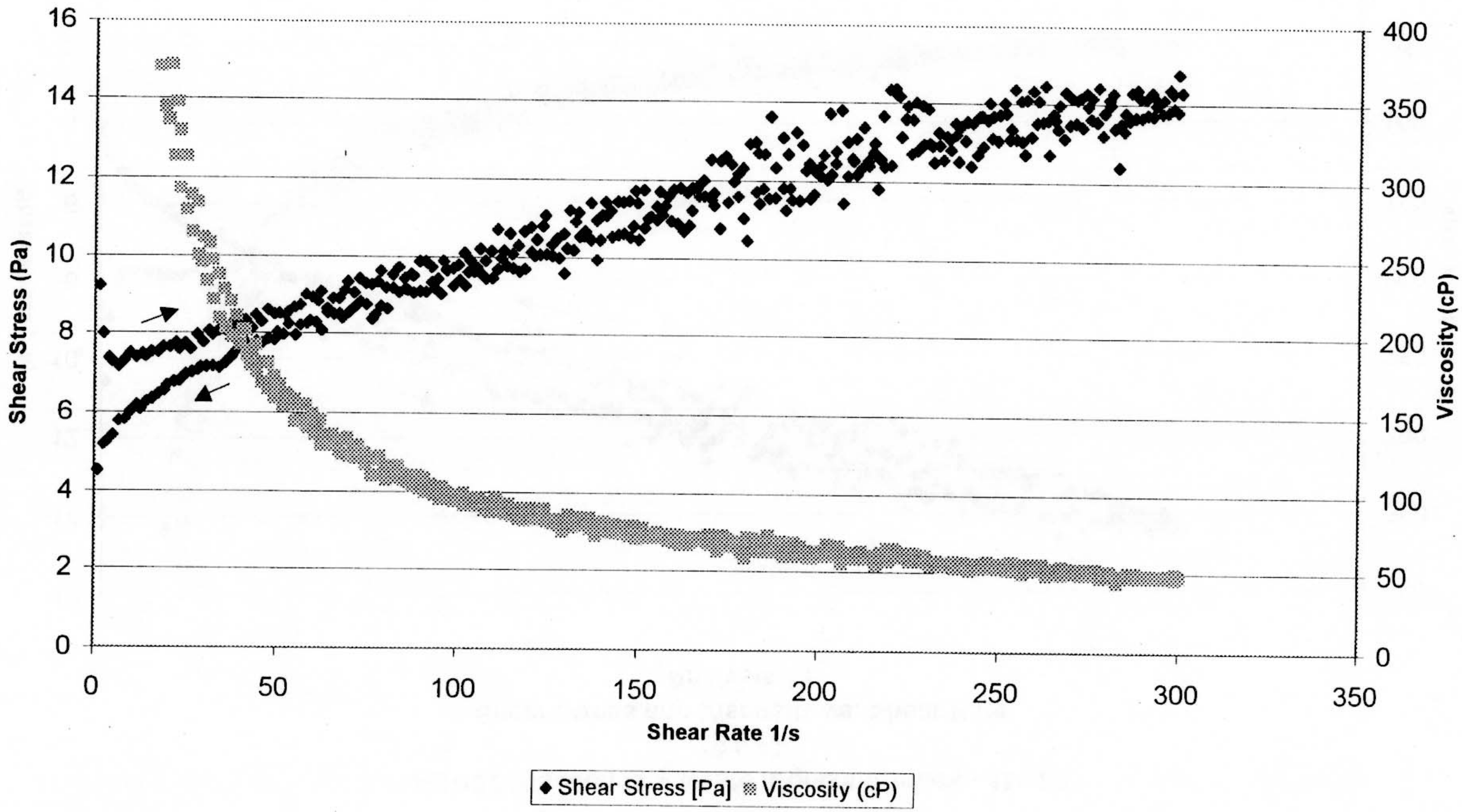
Measurement performed with a Haake M5 and SV I sensor

AZ-102 w/secondary waste & glass formers - 5wt%
@ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

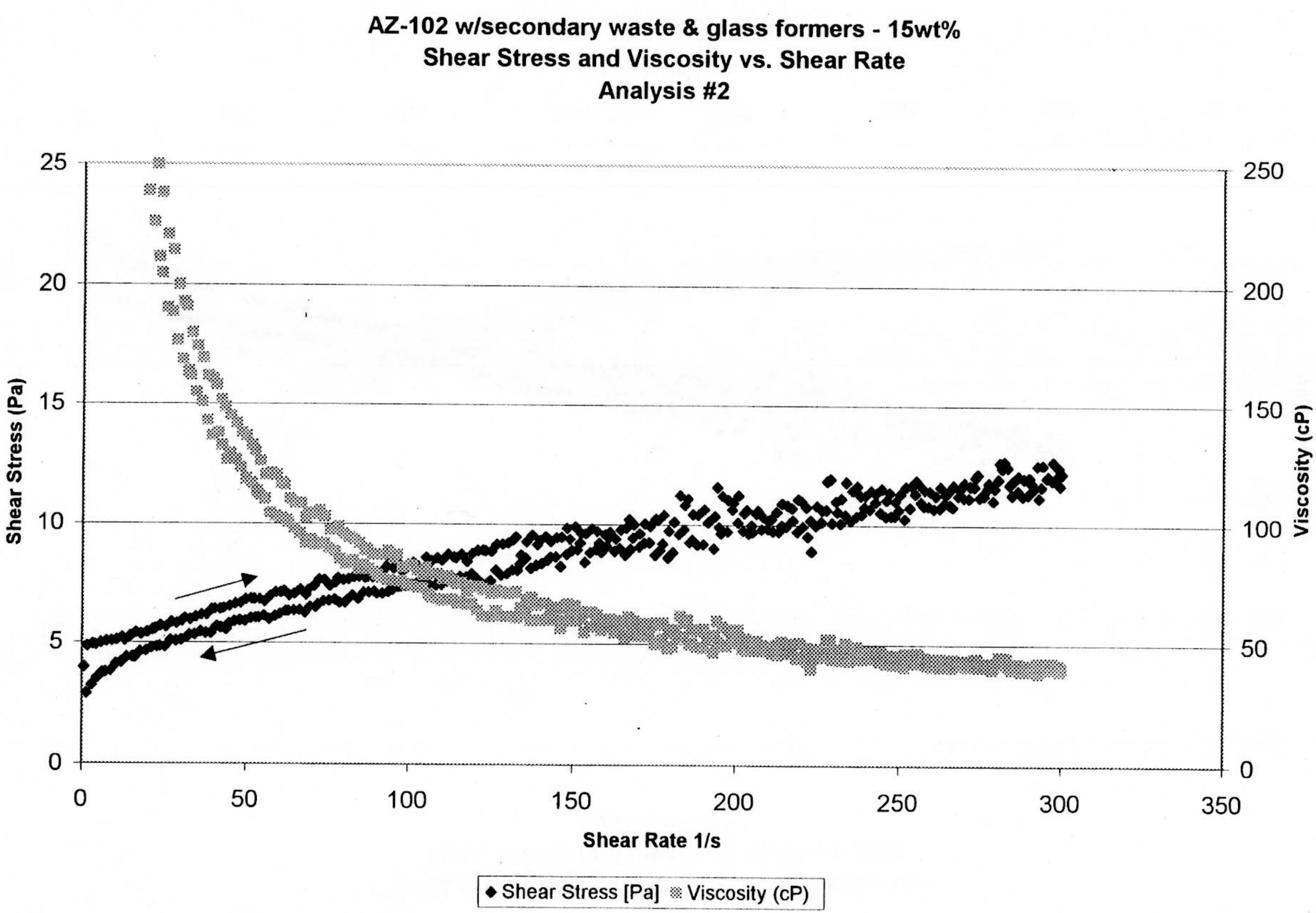


Measurement performed with a Haake M5 and SV I sensor

AZ-102 w/secondary waste & glass formers - 15wt%
@ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

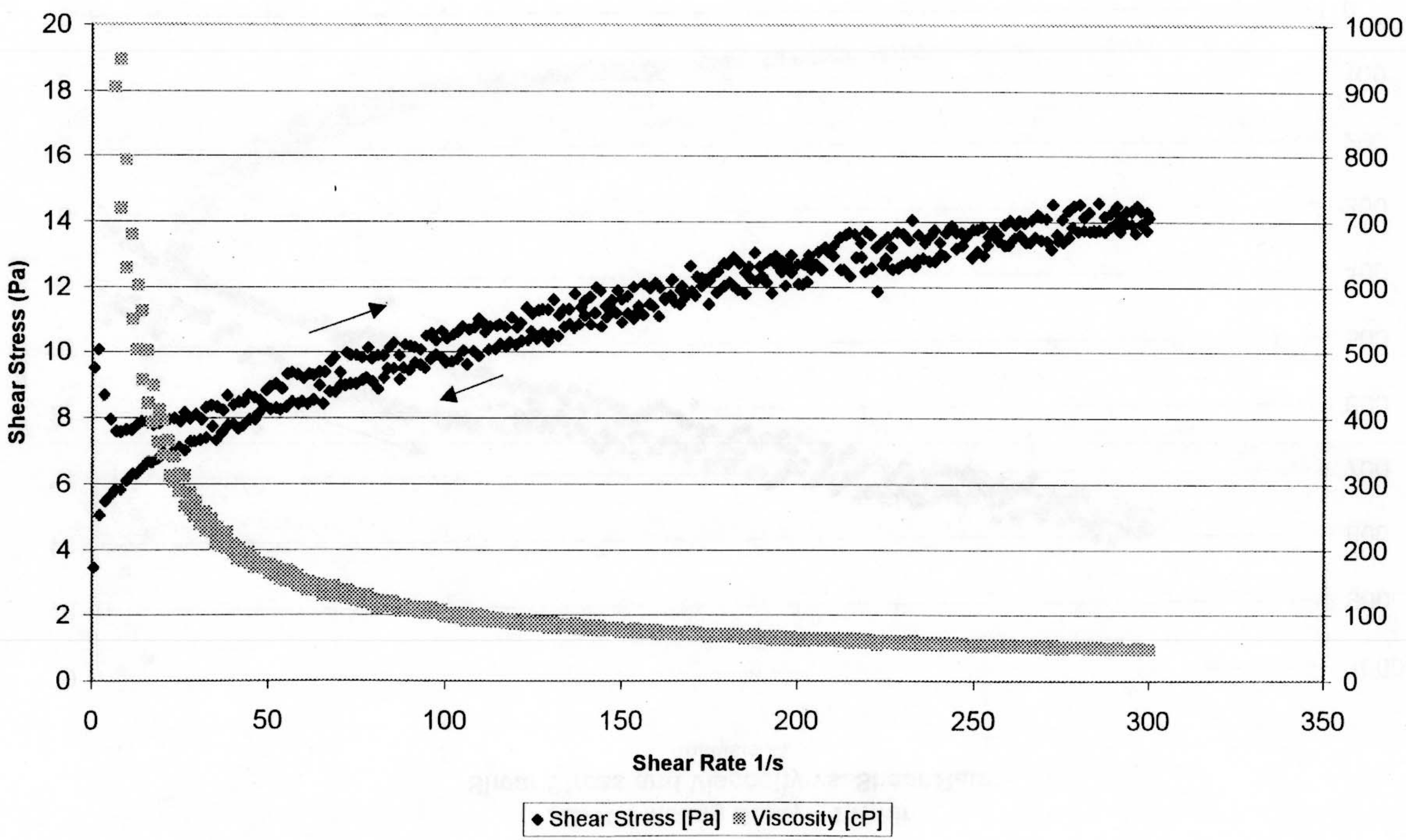


Measurement performed with a Haake M5 and SV I sensor



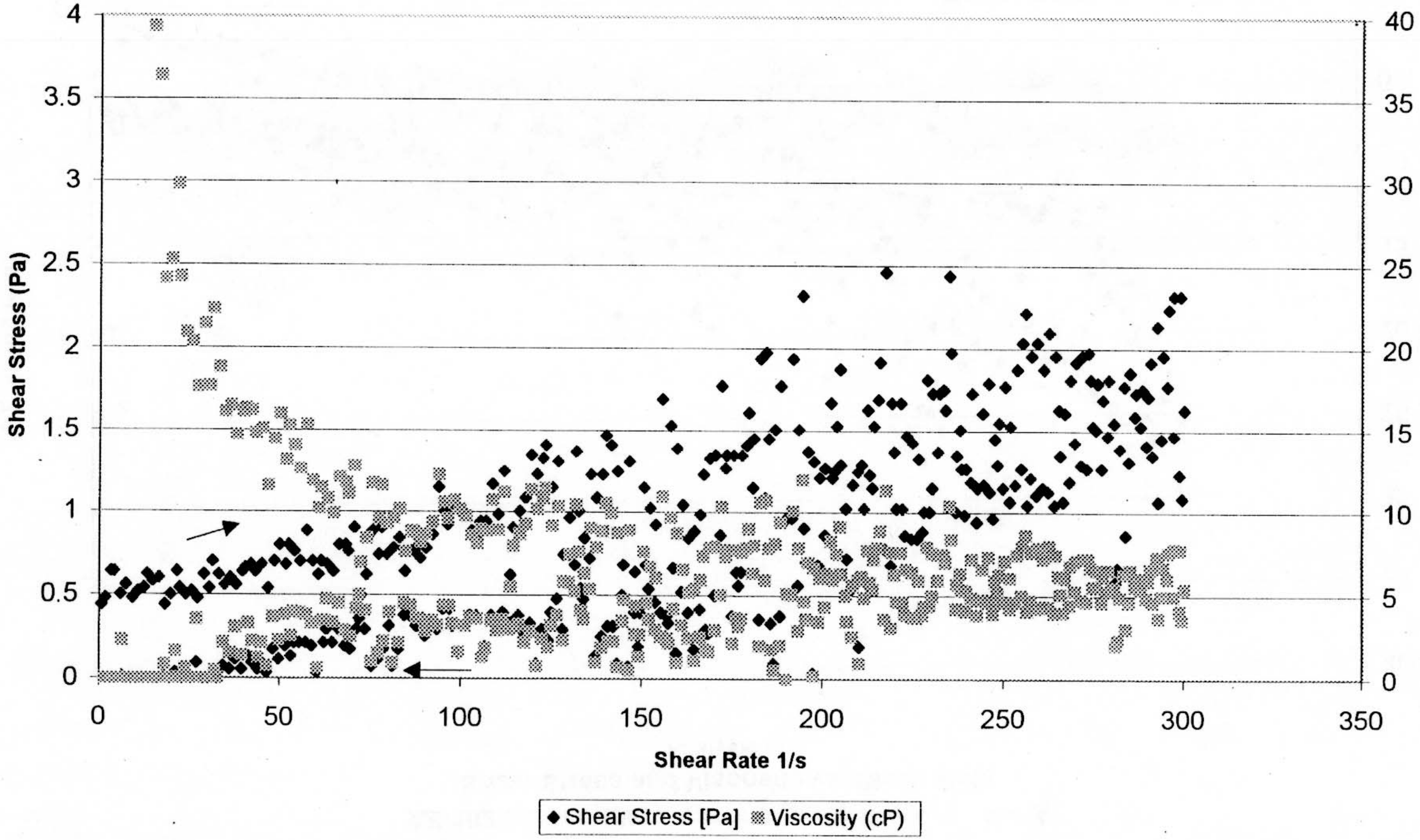
Measurement performed with a Haake M5 and SV I sensor

AZ-102 Mixing Study - 1 Hour
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



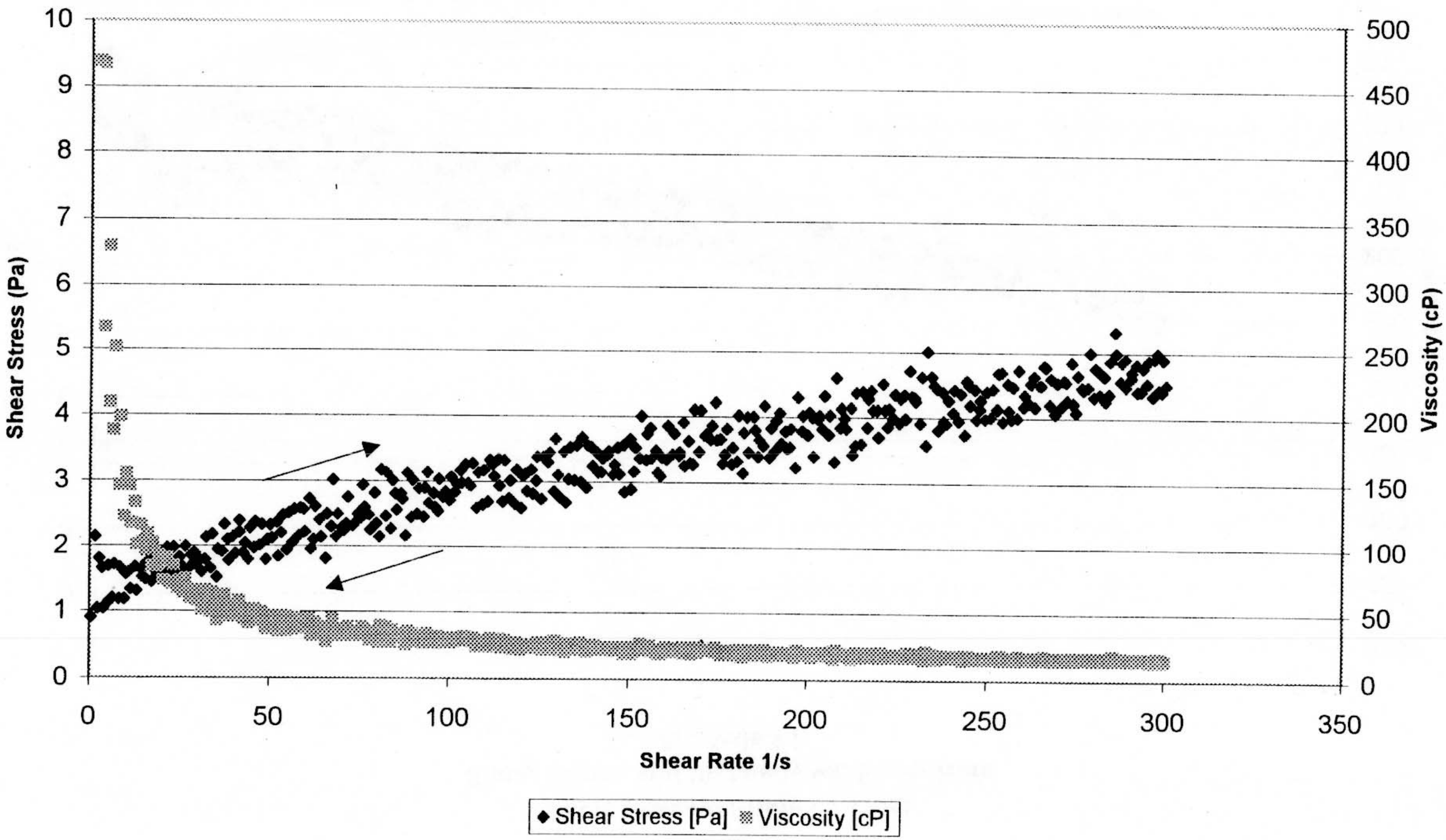
Measurement performed with a Haake M5 and SV I sensor

AZ-102 w/secondary waste & glass formers - 5wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



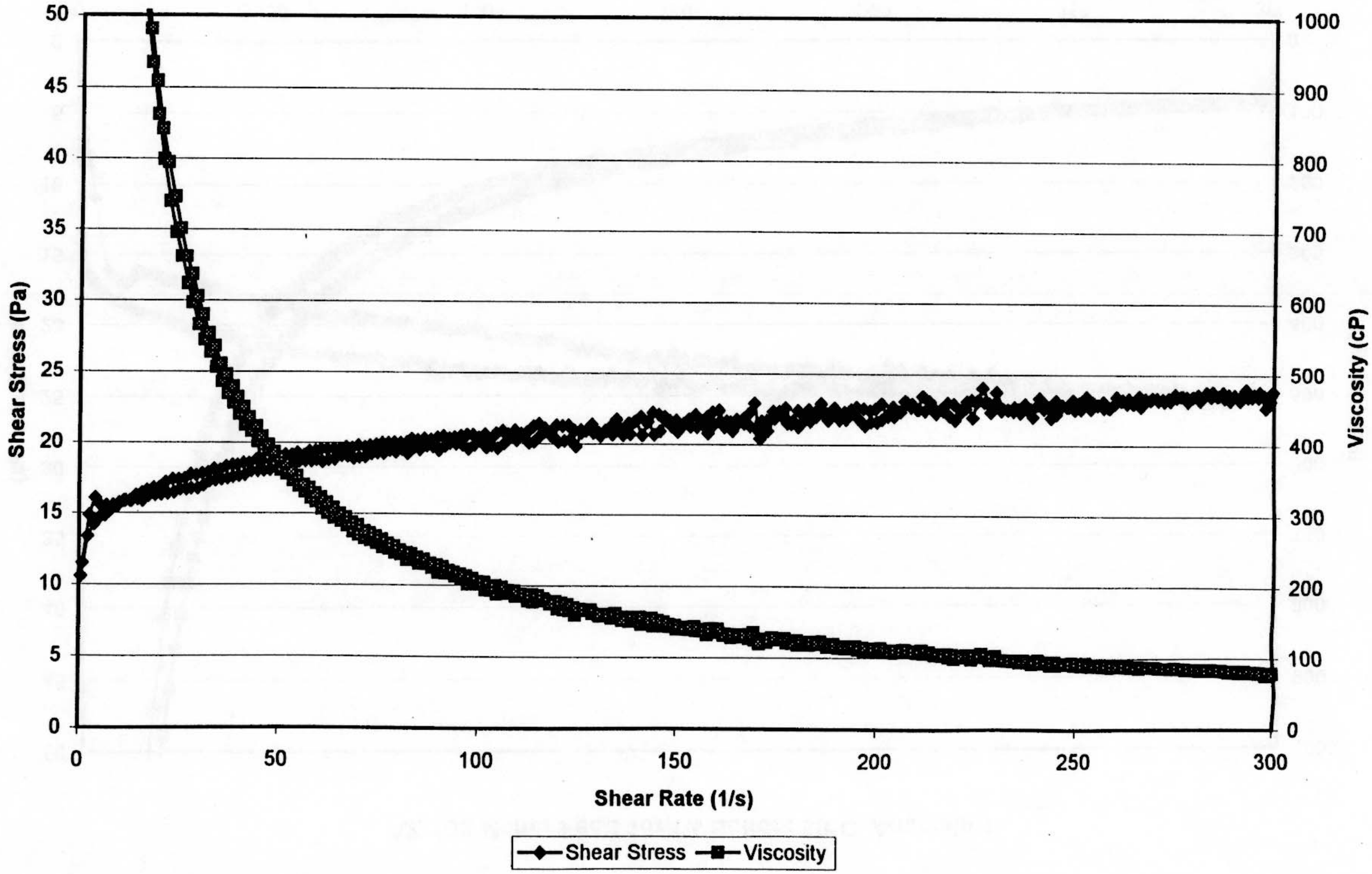
Measurement performed with a Haake M5 and SV I sensor

AZ-102 Mixing Study - 1 Week
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



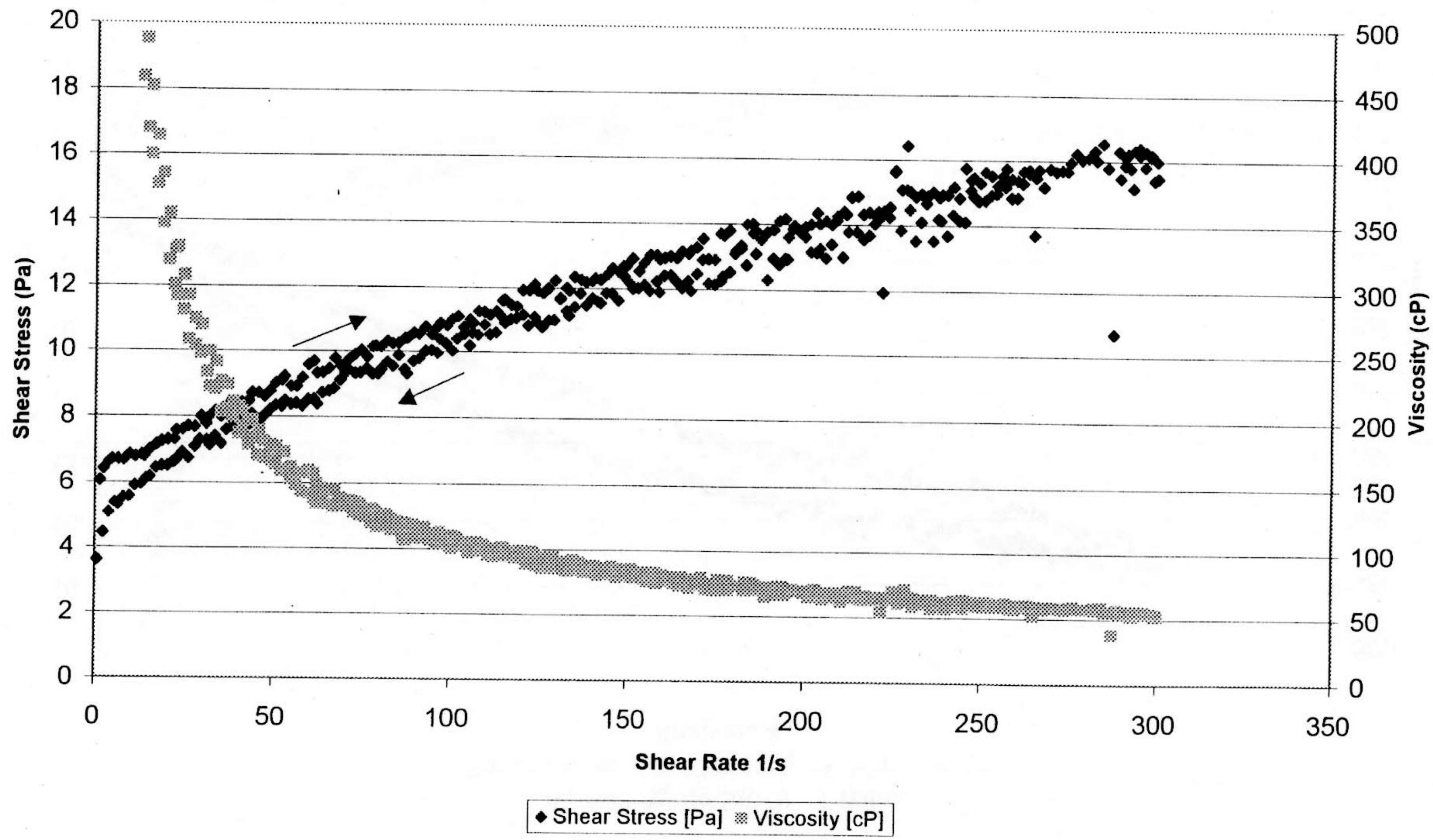
Measurement performed with a Haake M5 and SV I sensor

AZ-102 Melter Feed 15wt% Solids: 50°C, Analysis 2



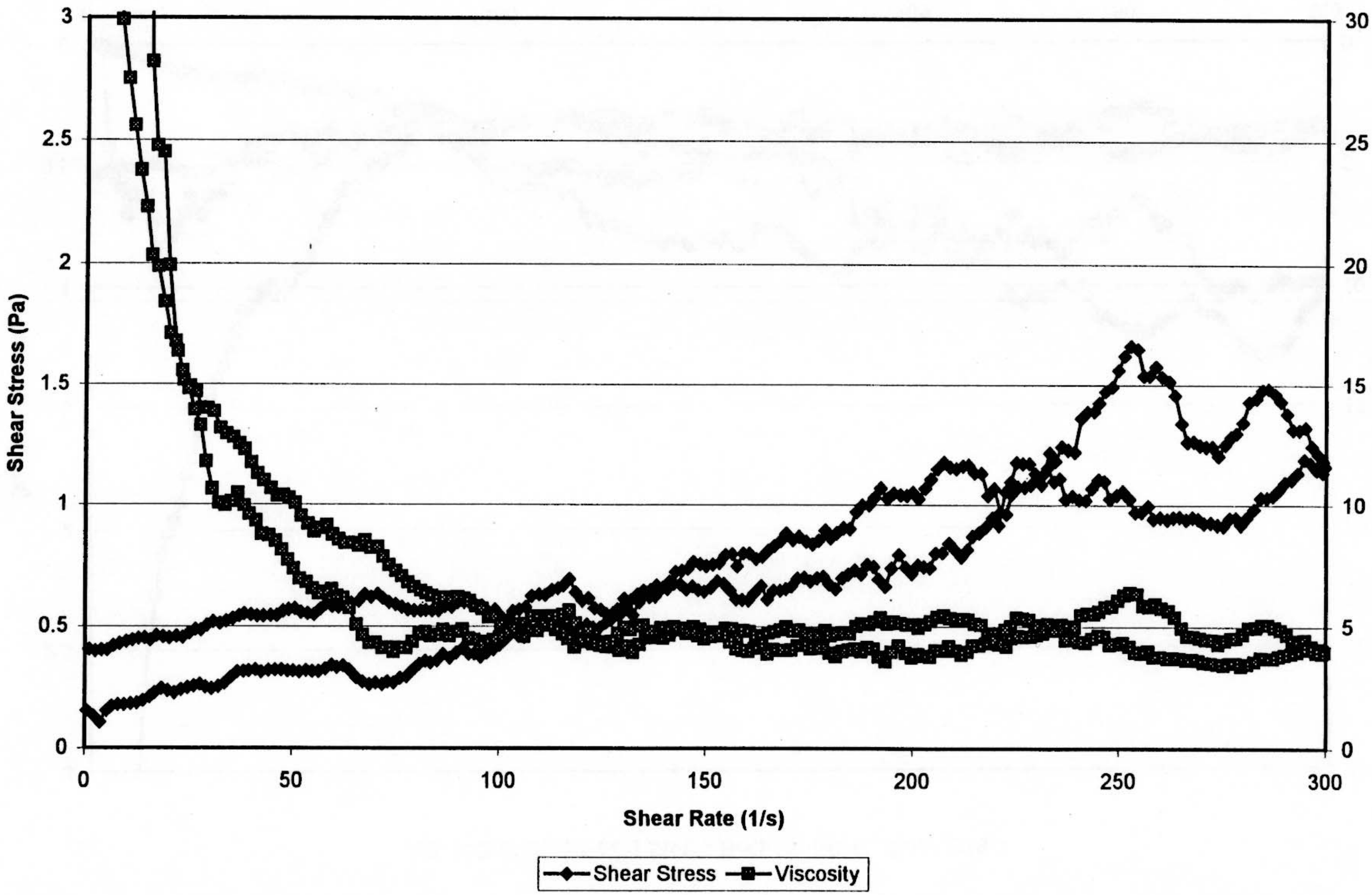
Haake M5 Viscometer with SV1 Geometry

AZ-102 Aging Study - 1 Week
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

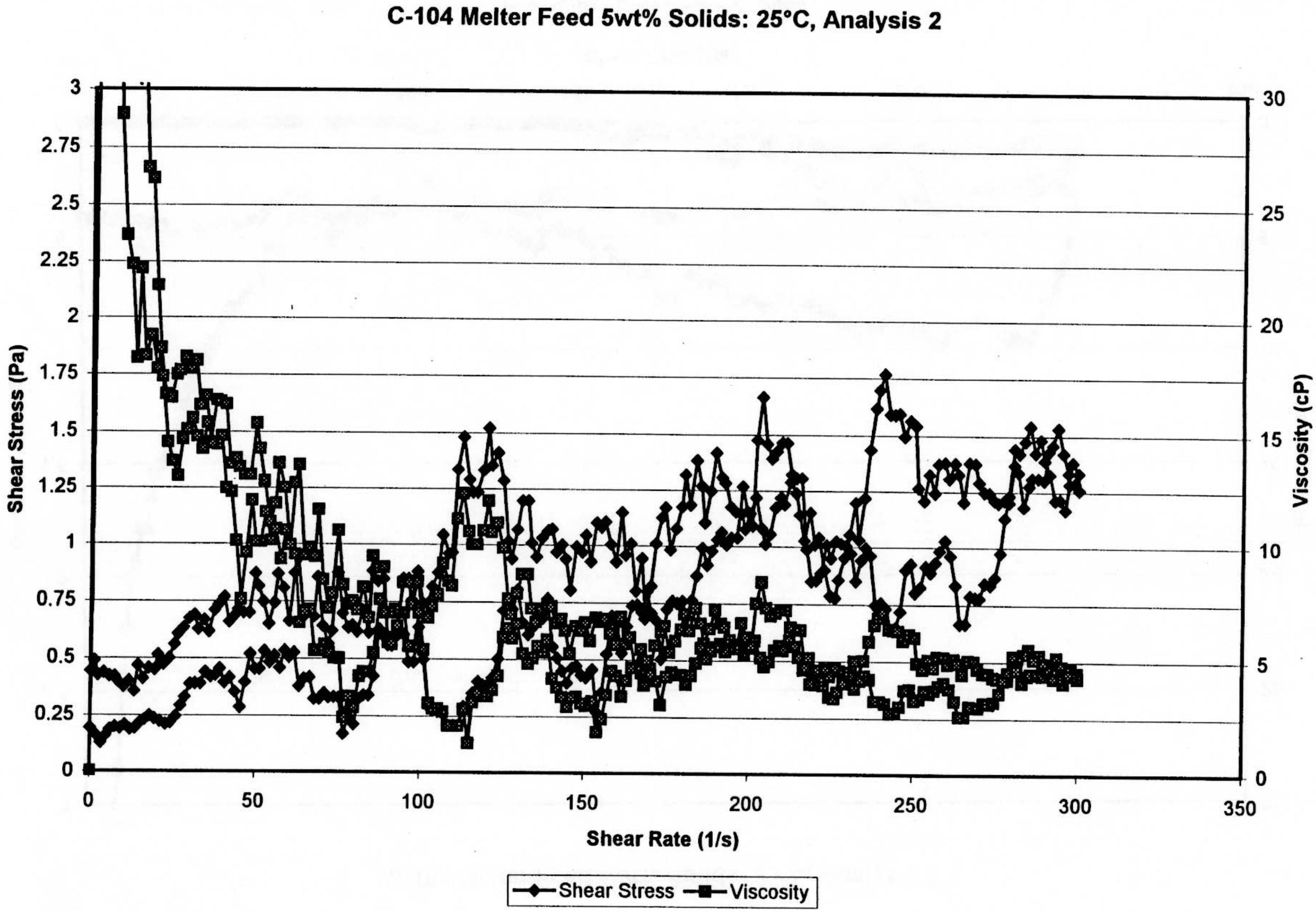


Measurement performed with a Haake M5 and SV I sensor

AZ-102 Melter Feed 5wt% Solids: 50°C, Analysis 2

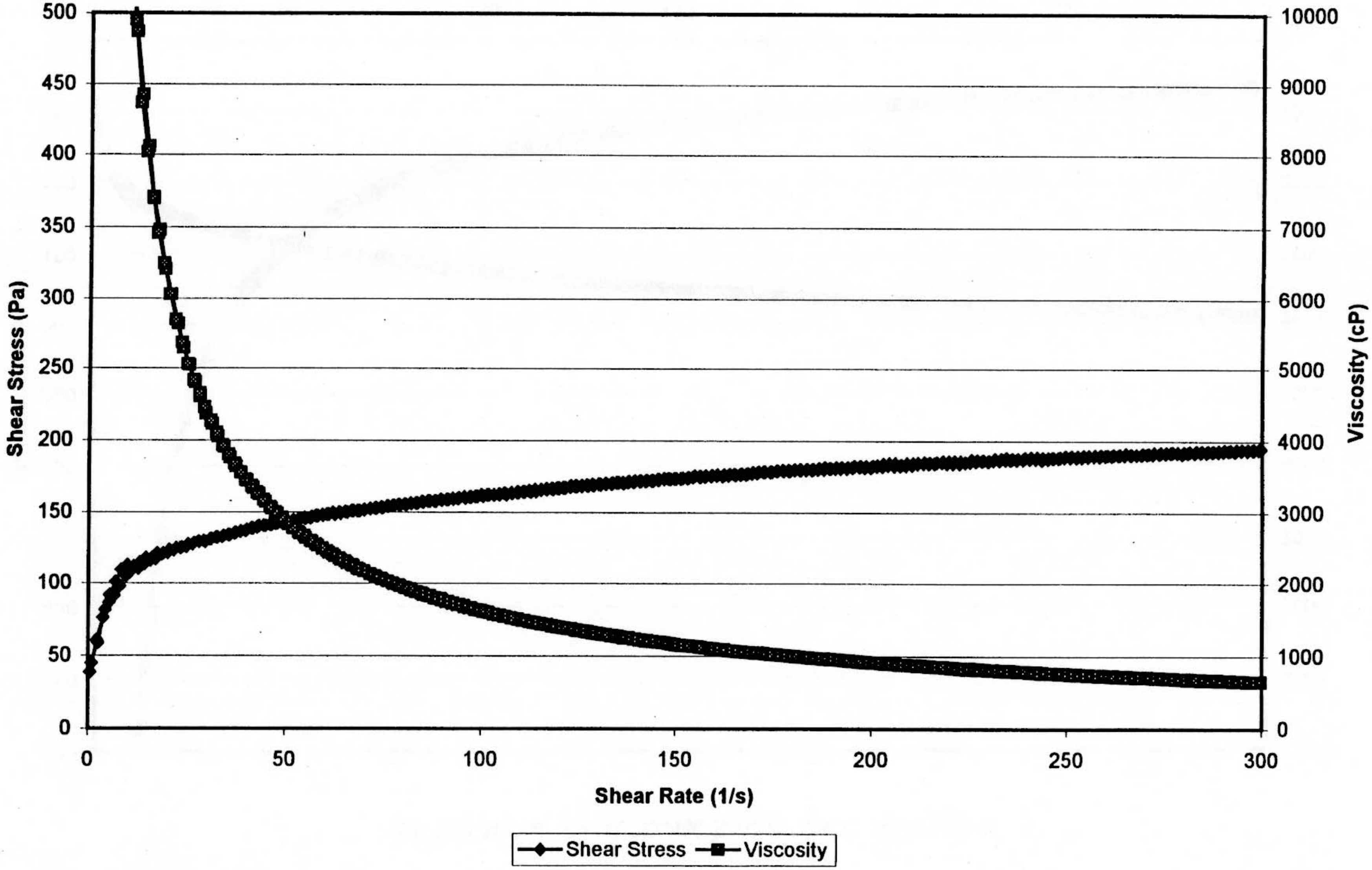


Haake M5 Viscometer with SV1 Geometry



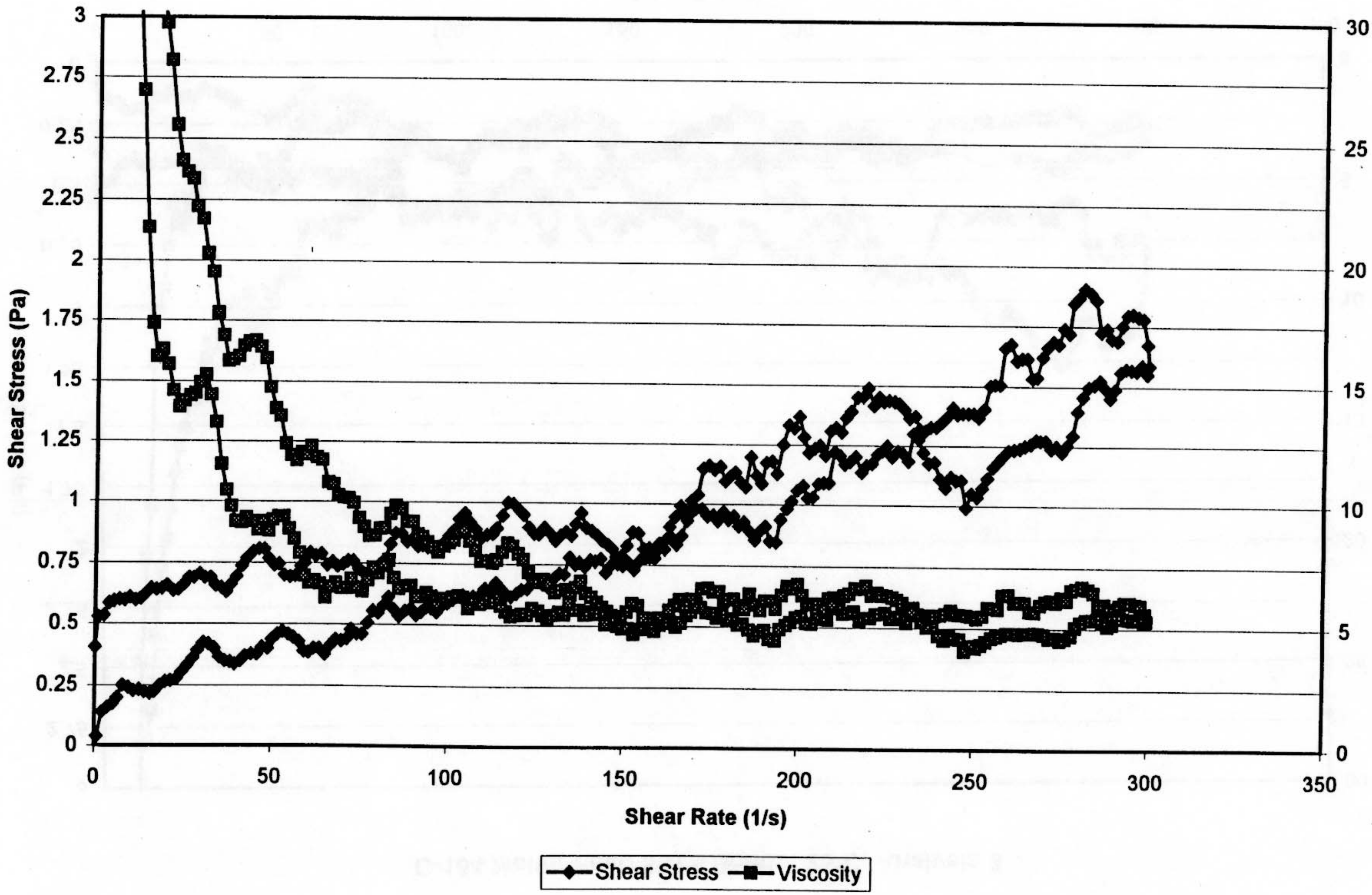
Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry

AZ-102 Melter Feed 25wt% Solids: 25°C, Analysis 3



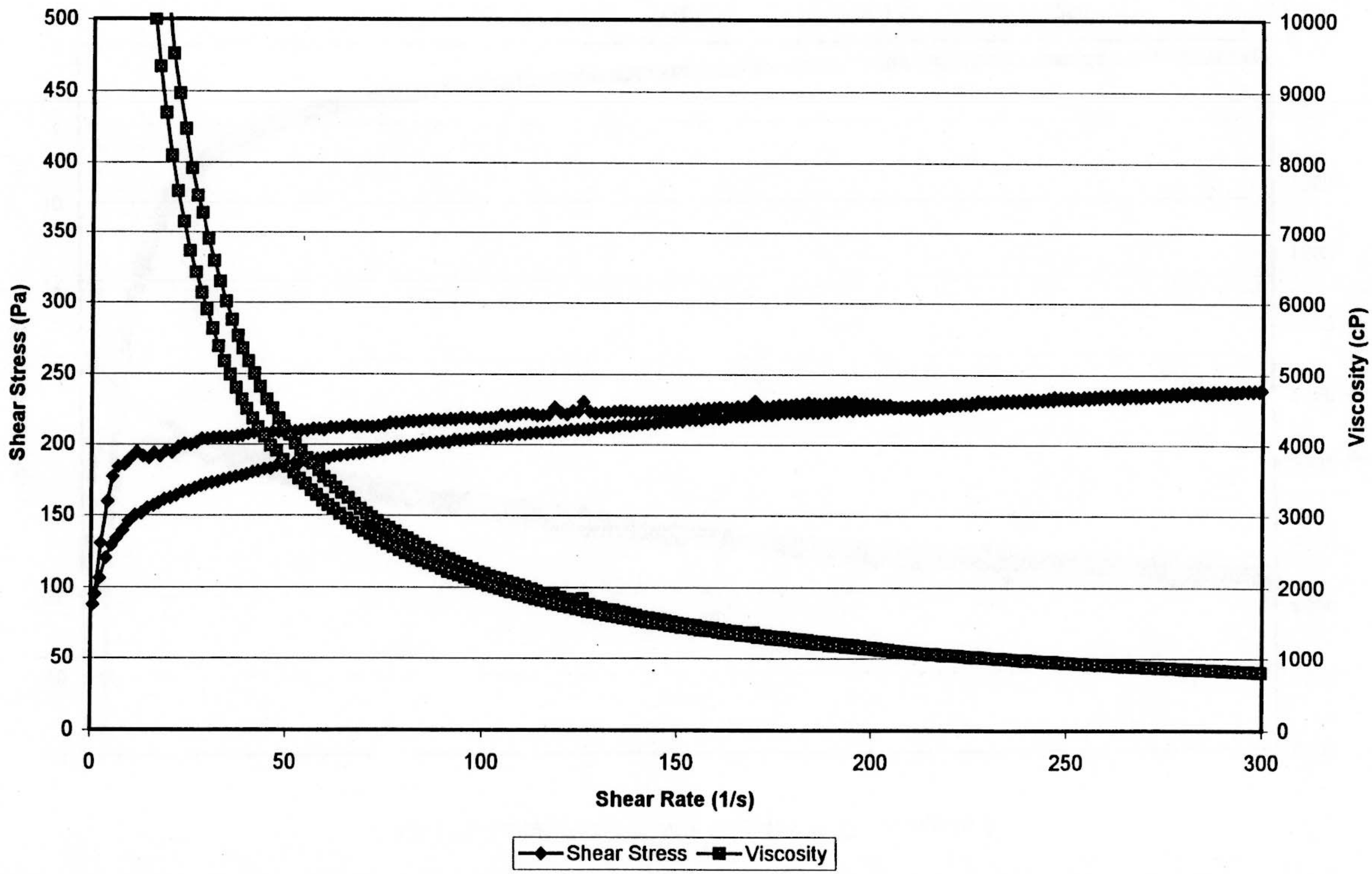
Haake M5 Viscometer with SV1 Geometry

C-104 Melter Feed 15wt% Solids: 25°C, Analysis 1



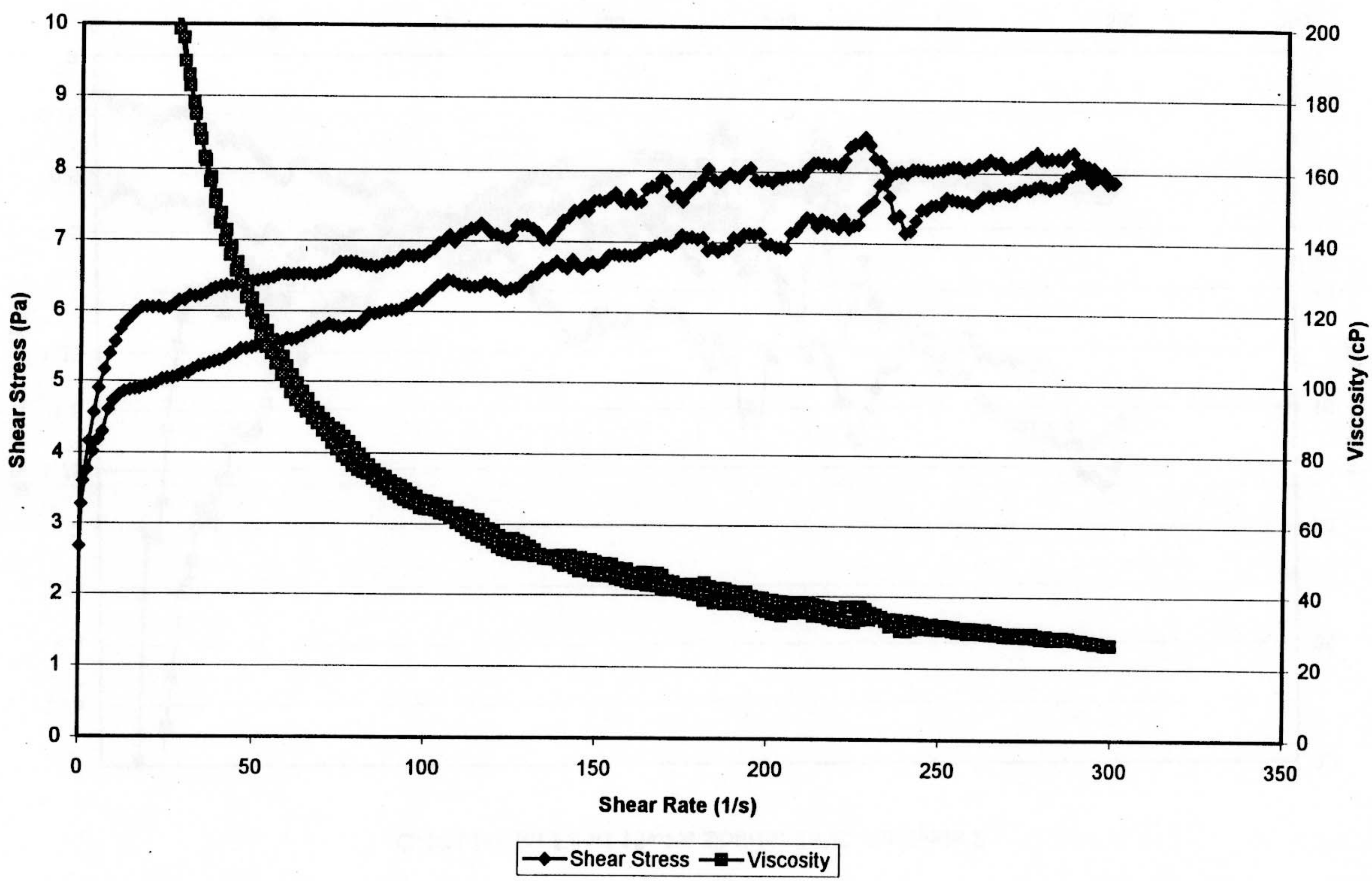
Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry

AZ-102 Melter Feed 25wt% Solids: 25°C, Analysis 1

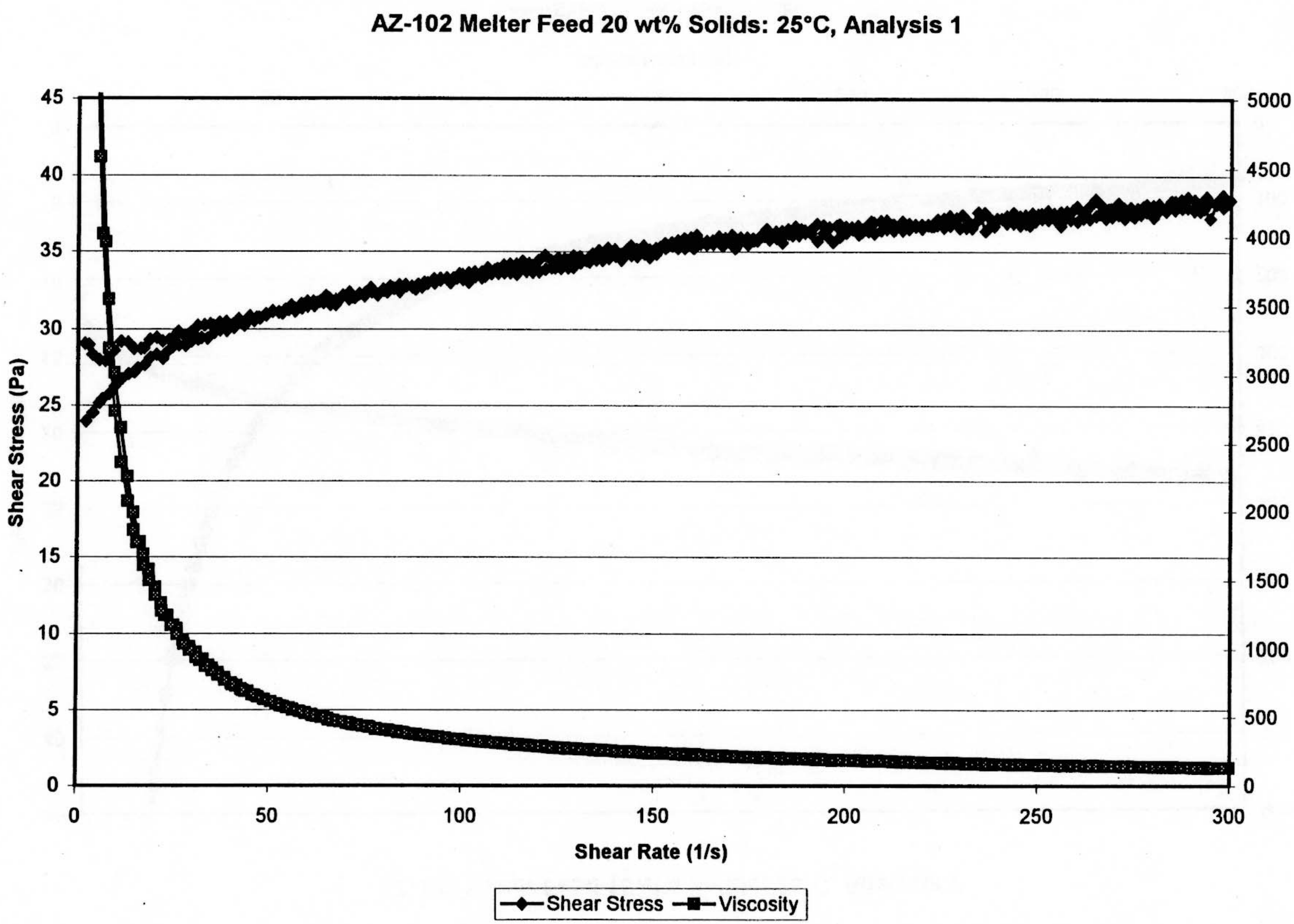


Haake M5 Viscometer with SV1 Geometry

C-104 Melter Feed 25wt% Solids: 25°C, Analysis 1

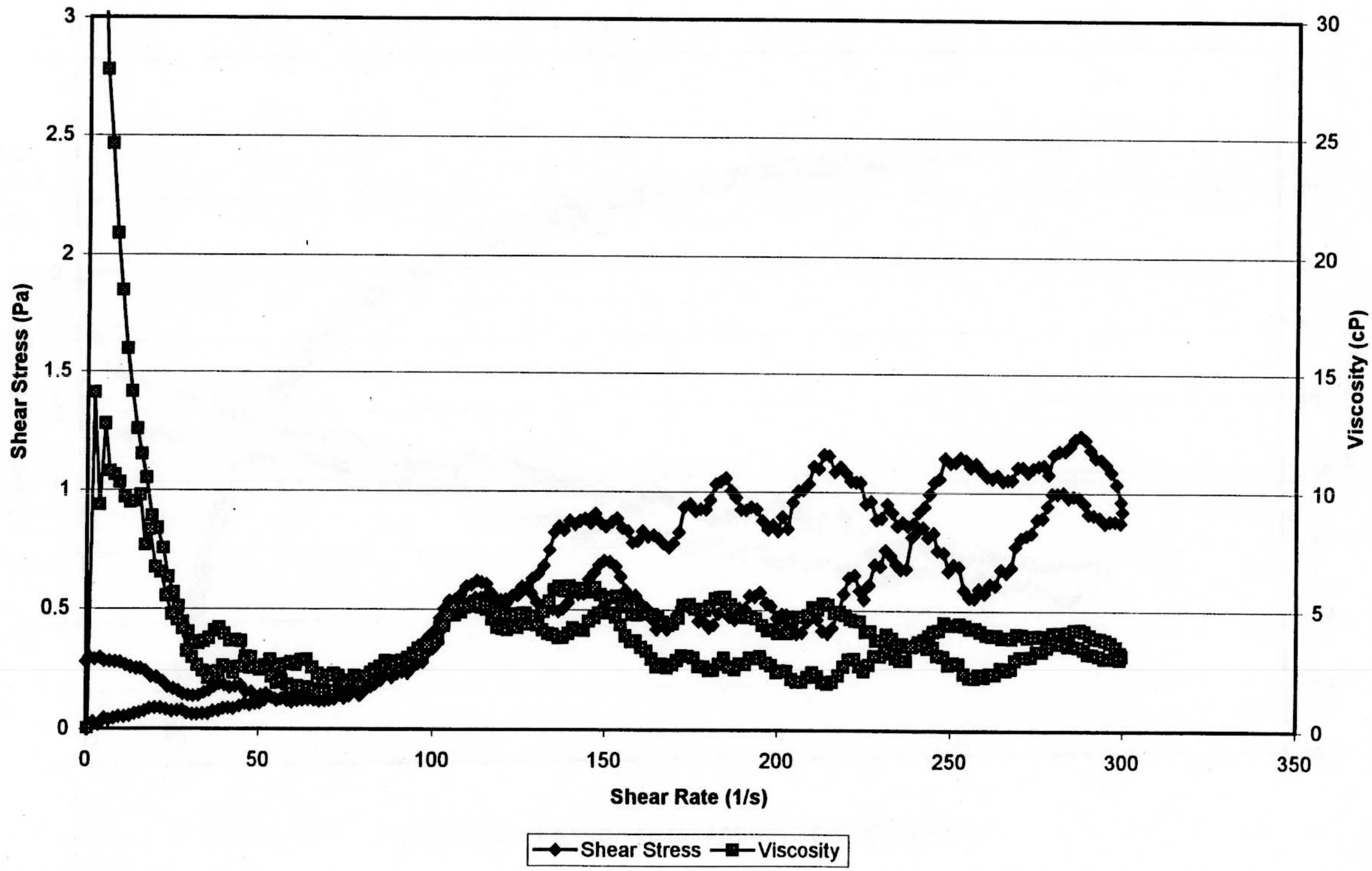


Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry



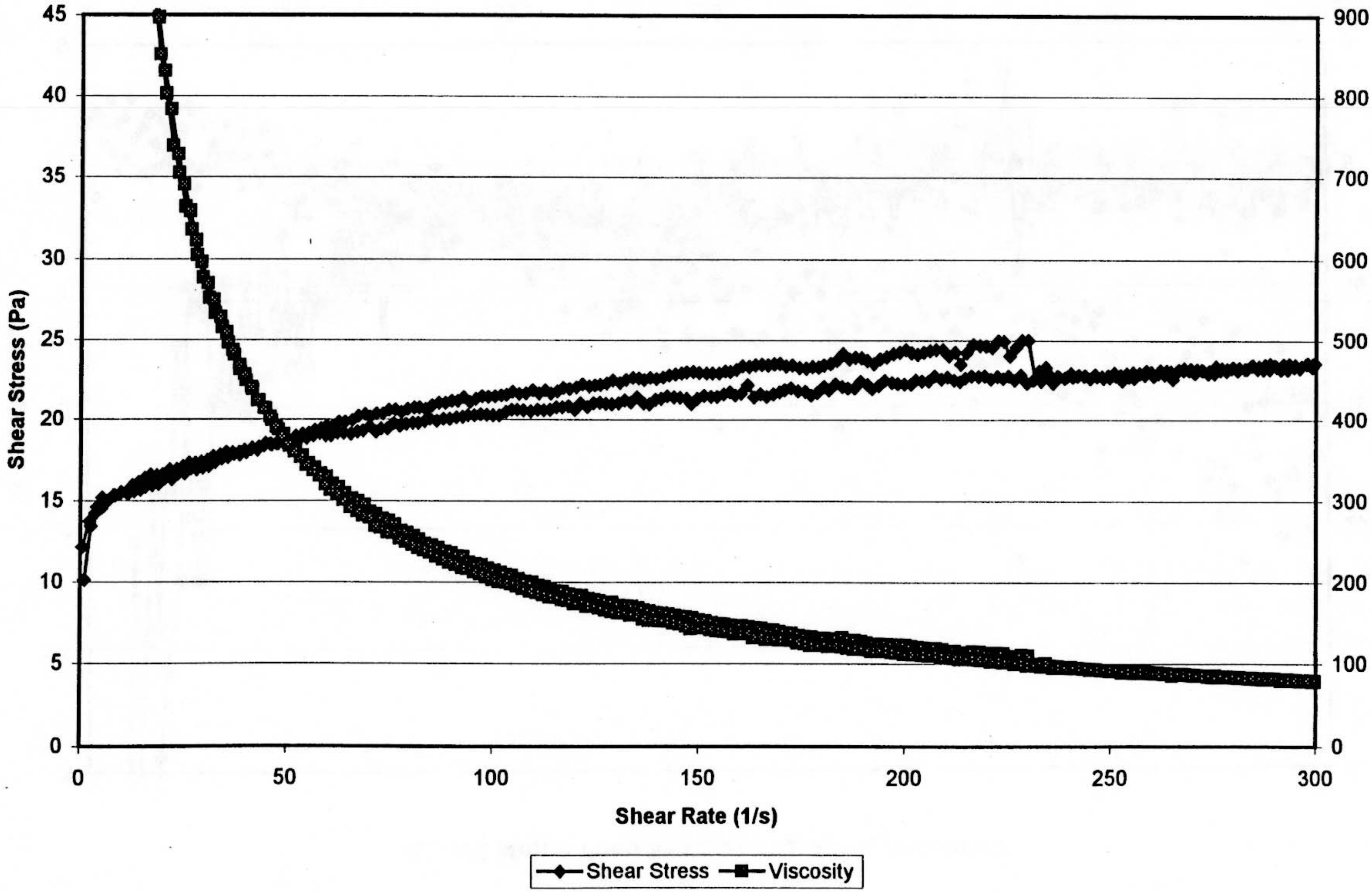
Haake M5 Viscometer with SV1 Geometry

C-104 Melter Feed 5wt% Solids: 50°C, Analysis 1



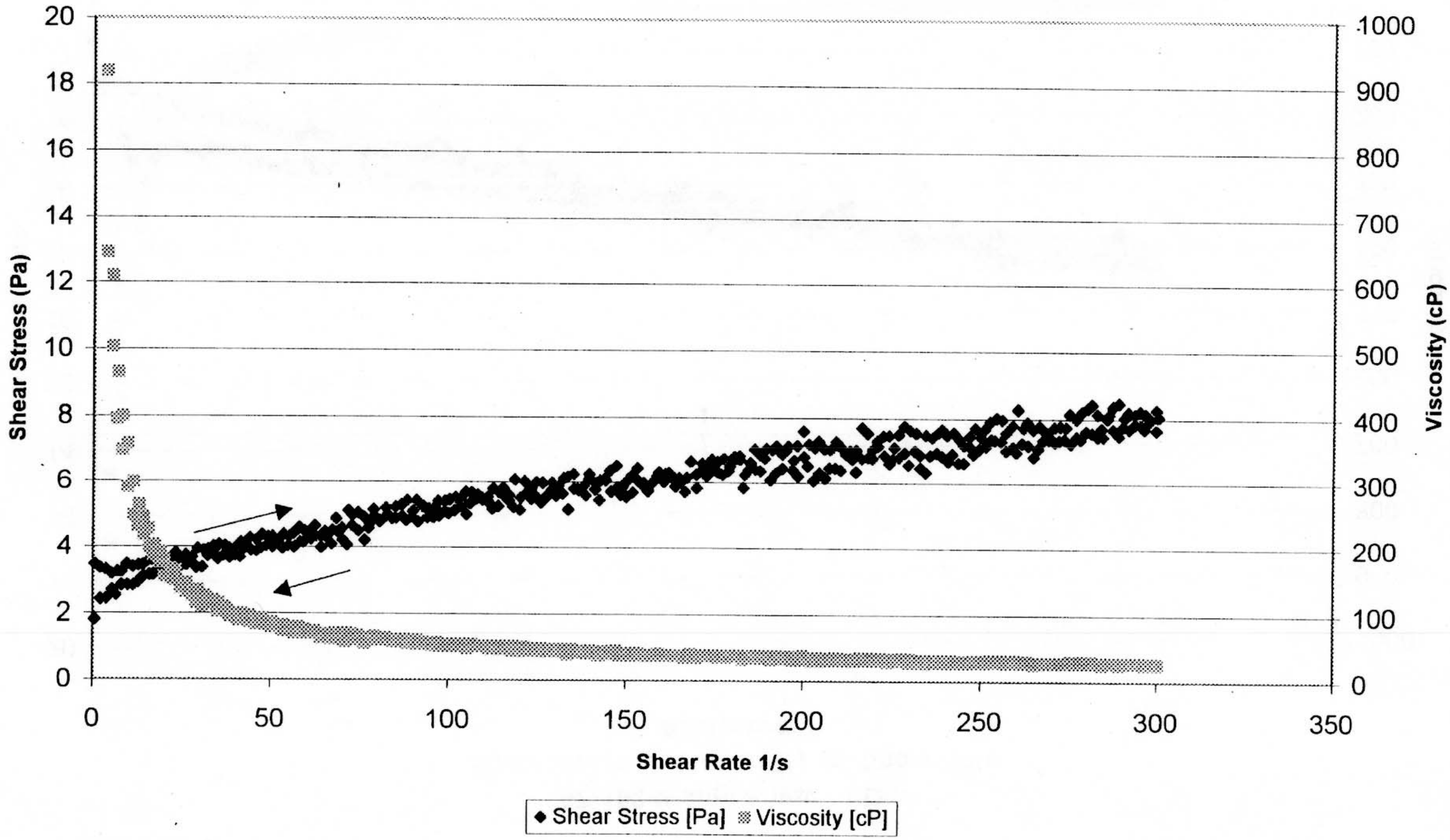
Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry

AZ-102 Melter Feed 15wt% Solids: 25°C, Analysis 1

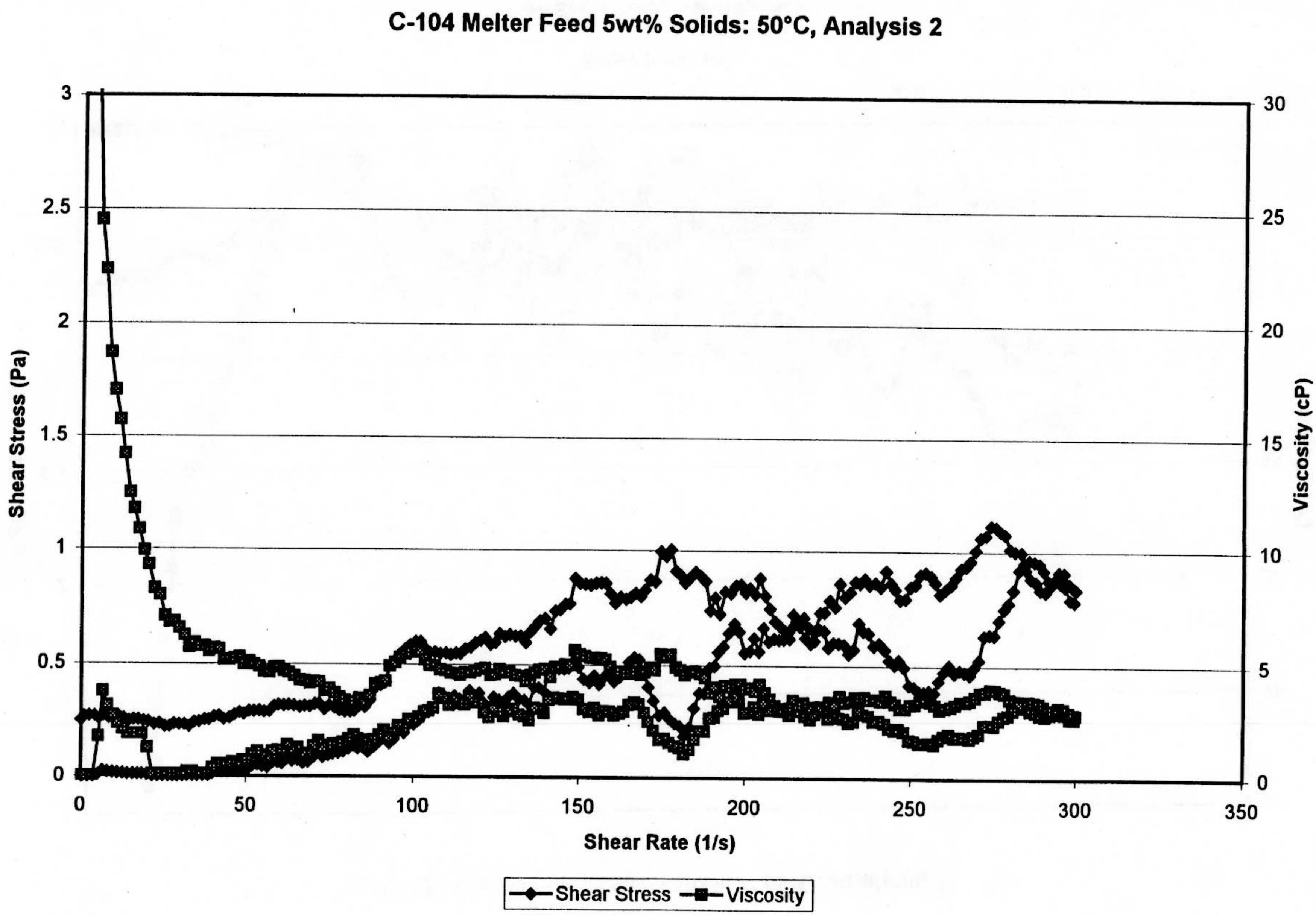


Haake M5 Viscometer with SV1 Geometry

AZ-102 Mixing Study - 1 Day
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

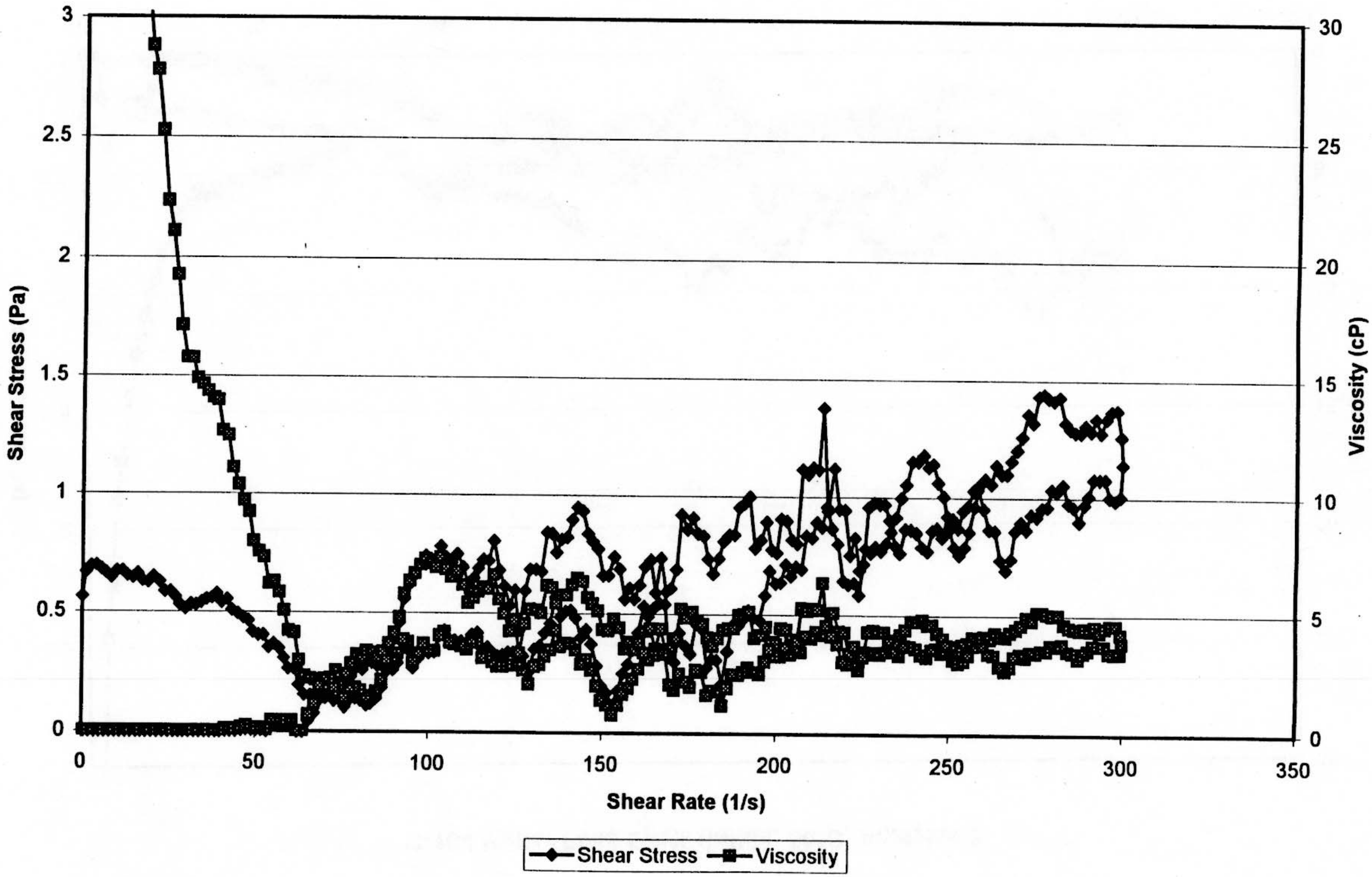


Measurement performed with a Haake M5 and SV I sensor



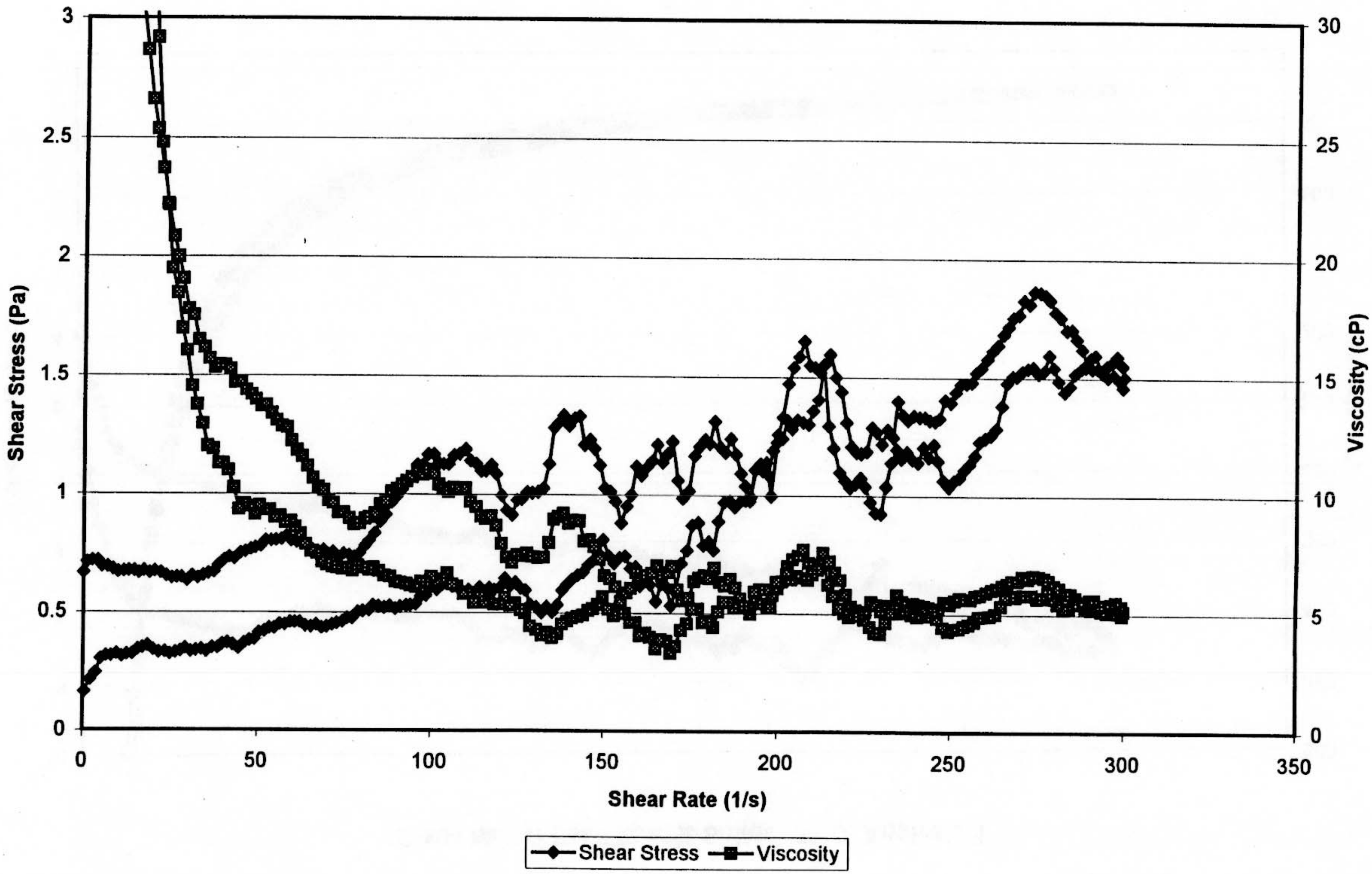
Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry

C-104 Melter Feed 15wt% Solids: 50°C, Analysis 1



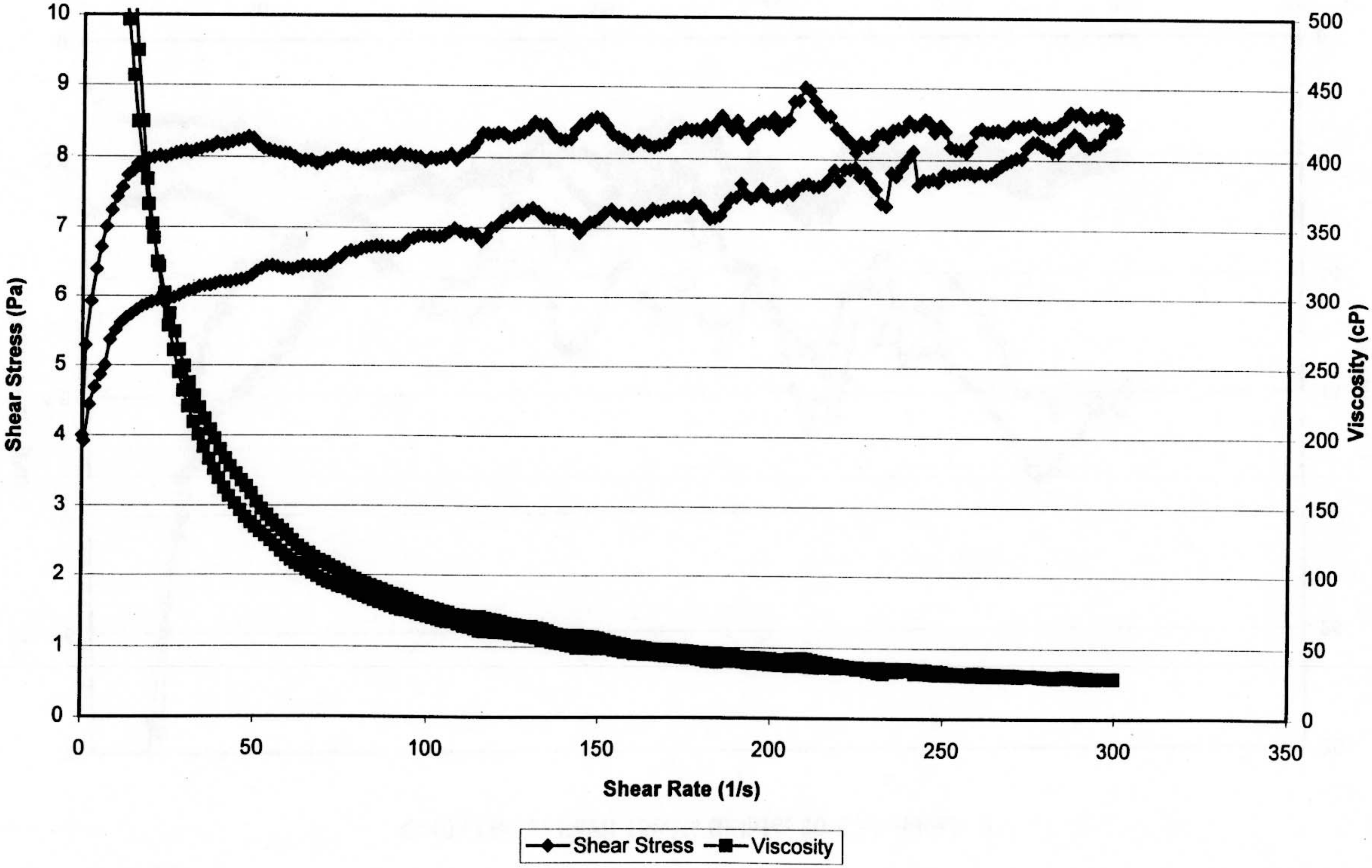
Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry

C-104 Melter Feed 15wt% Solids: 50°C, Analysis 2

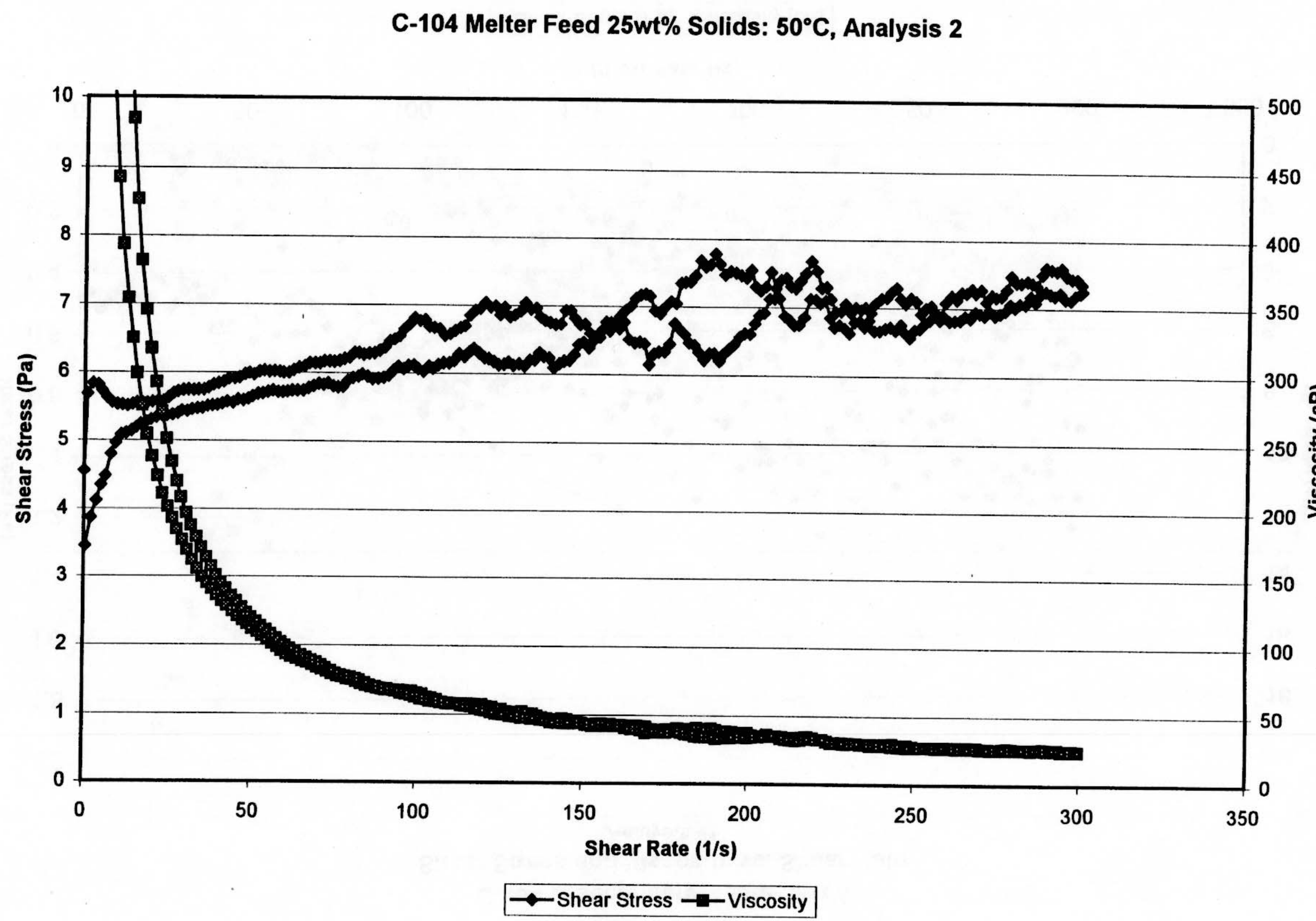


Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry

C-104 Melter Feed 25wt% Solids: 50°C, Analysis 1

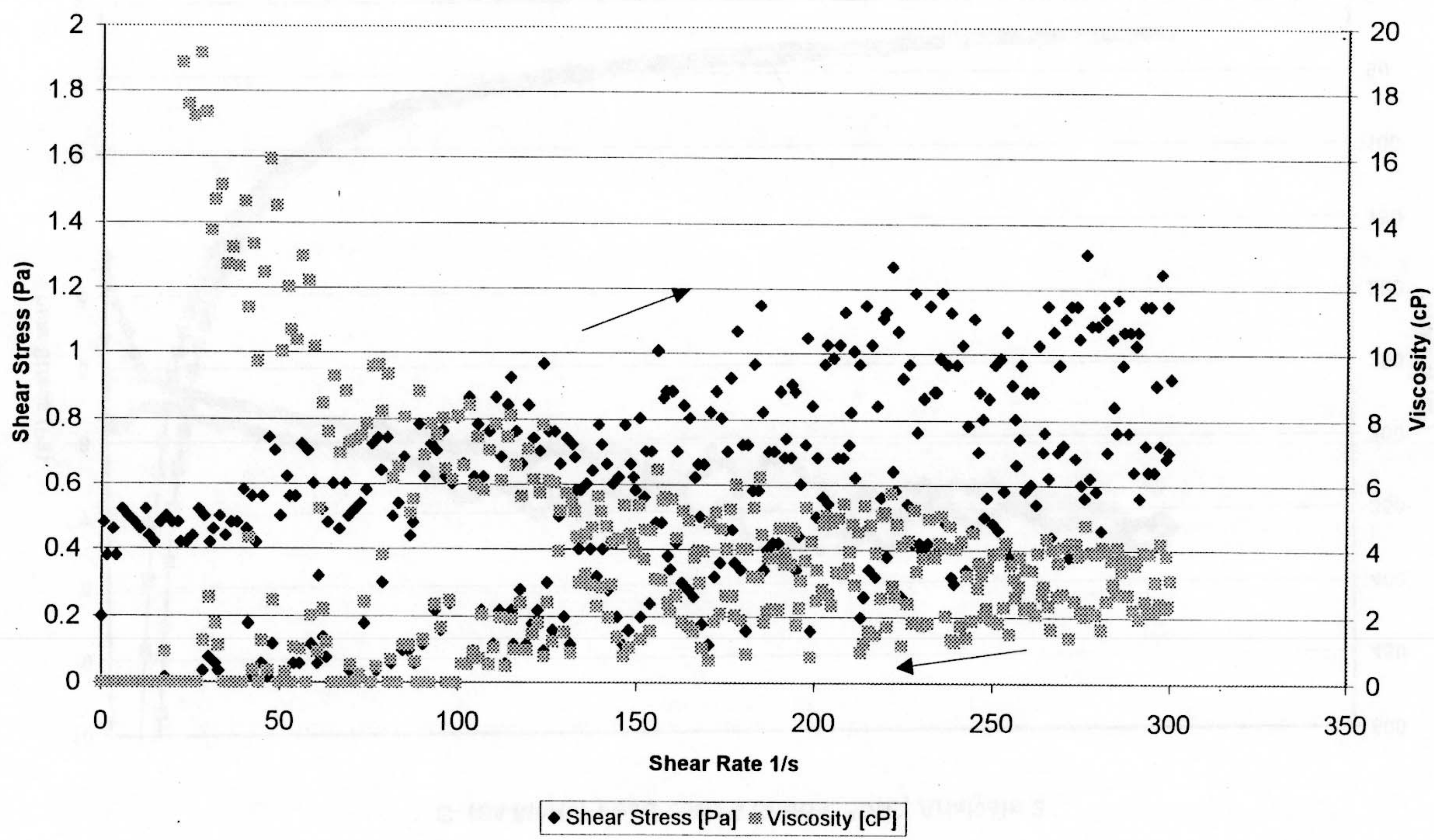


Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry

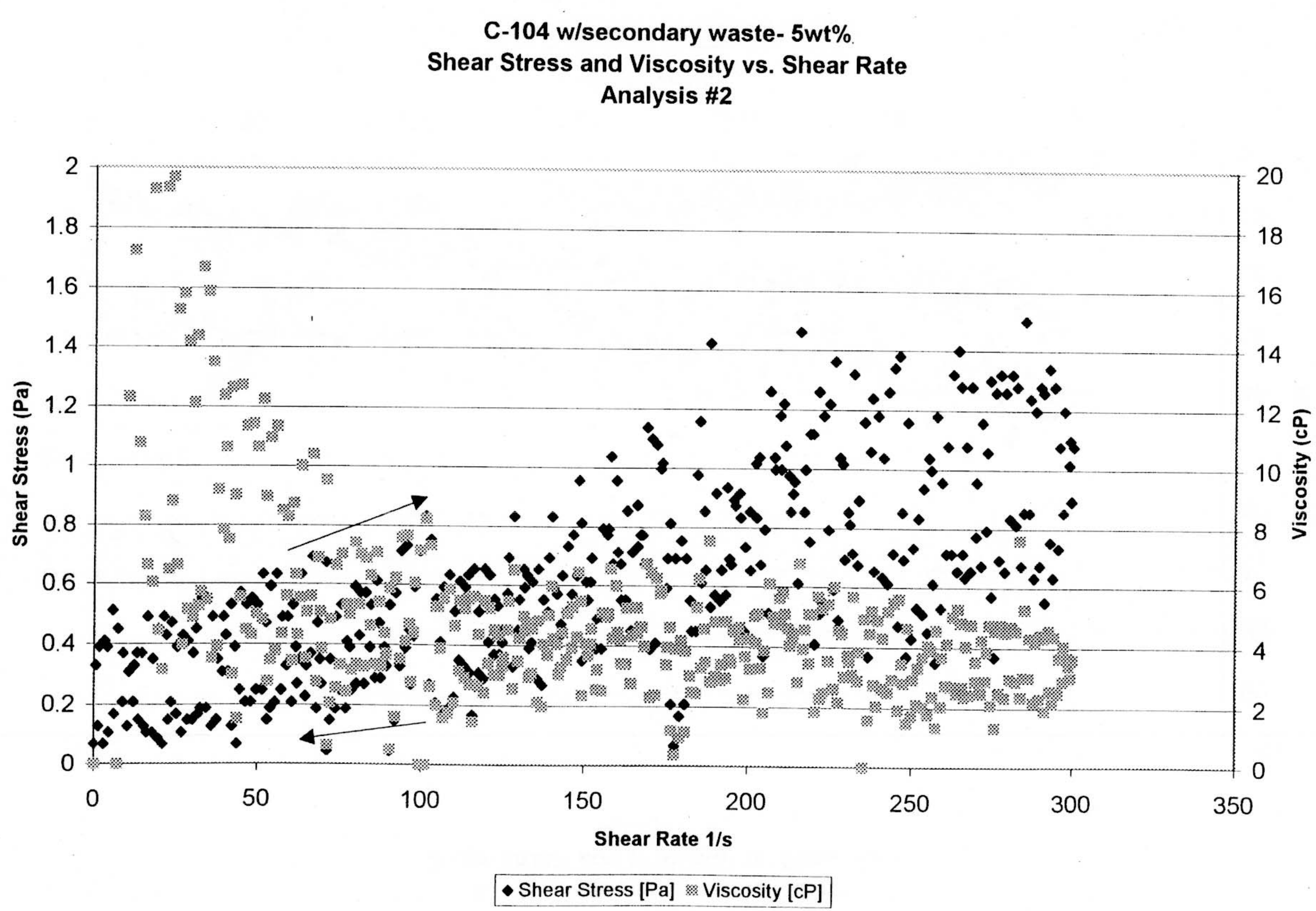


Analysis using M5 viscometer modified for hot cell operations: SV1 measuring geometry

C-104 w/secondary waste- 5wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #1

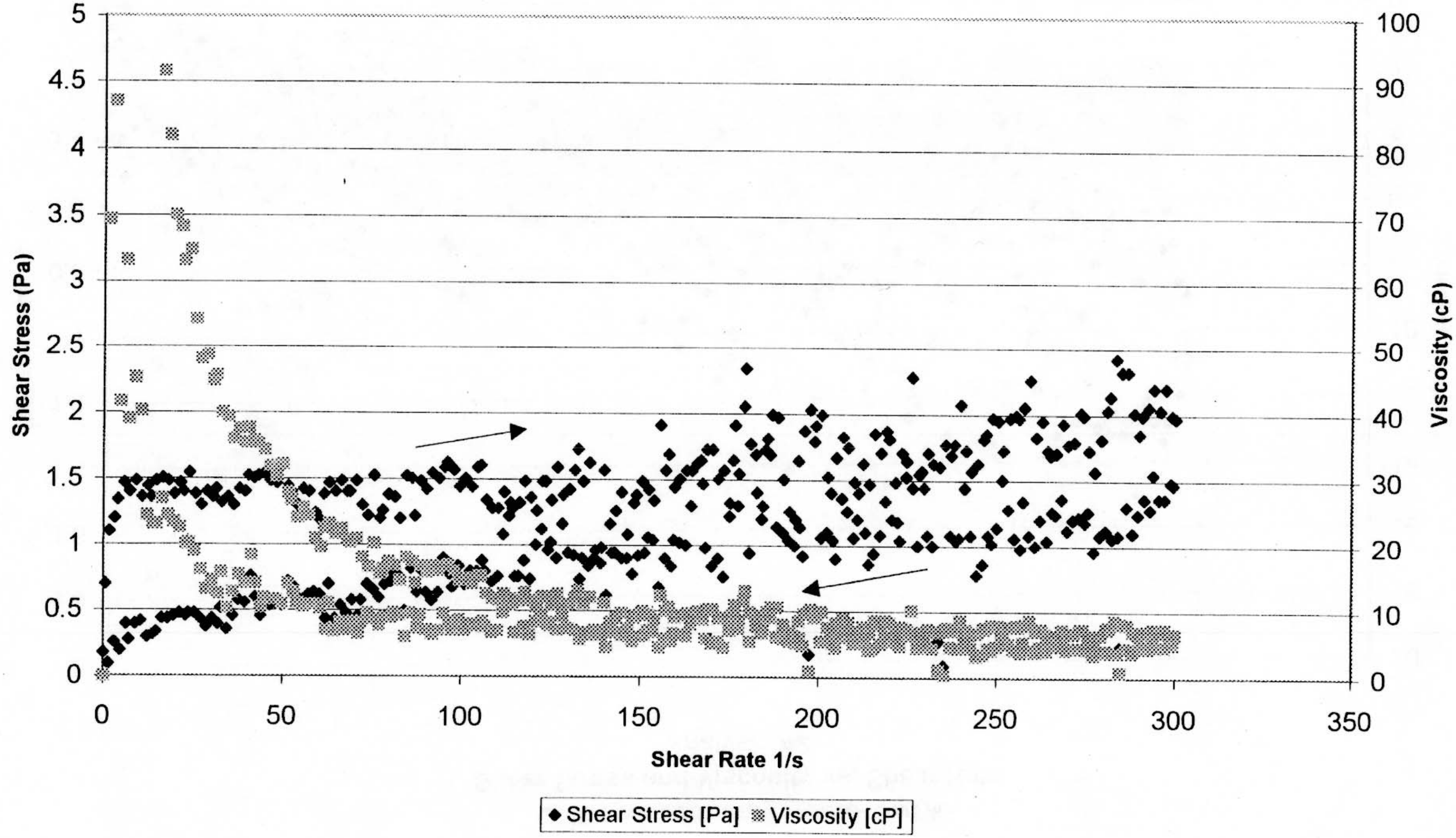


Measurement performed with a Haake M5 and SV I sensor



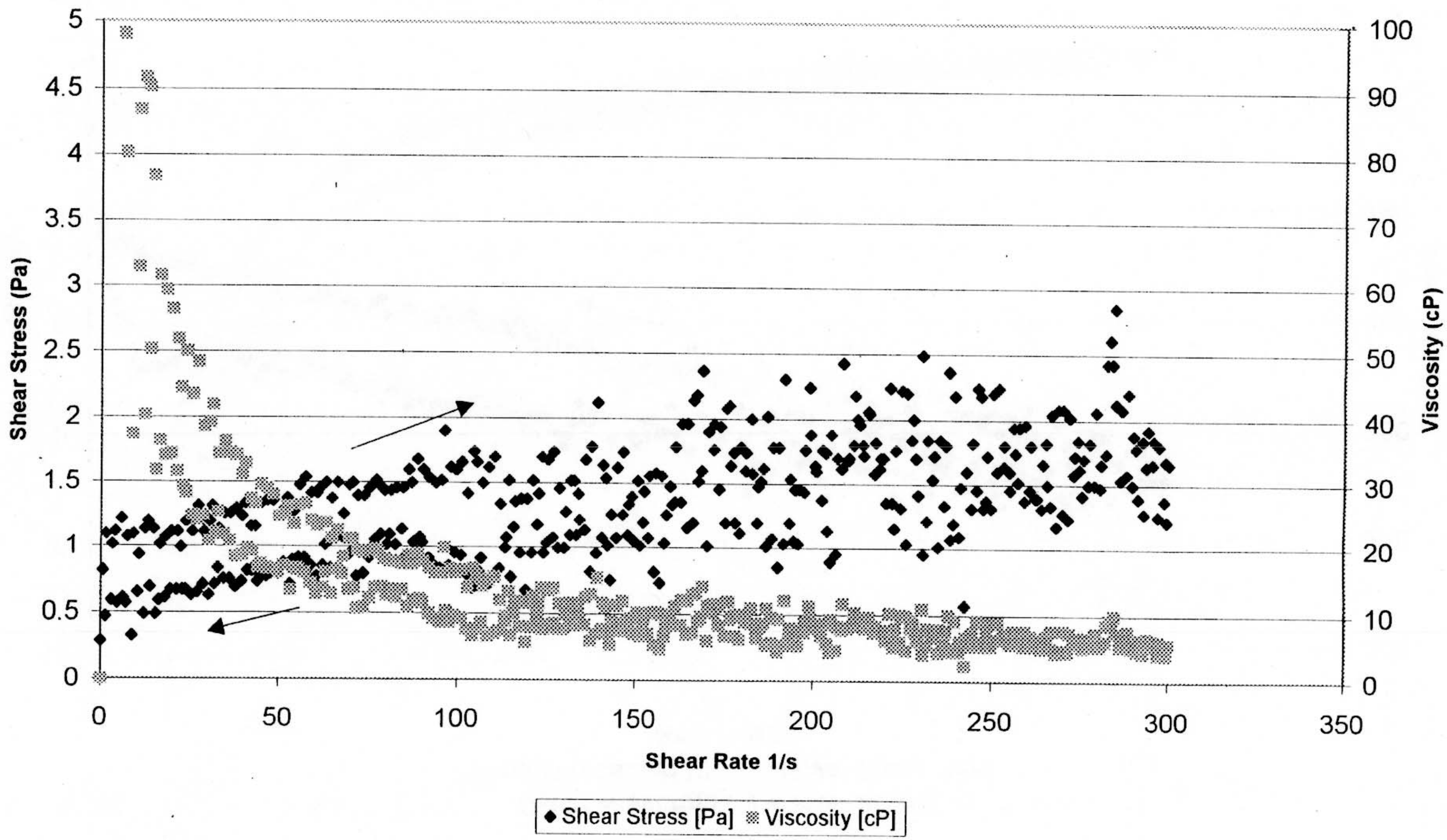
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste- 15wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #1



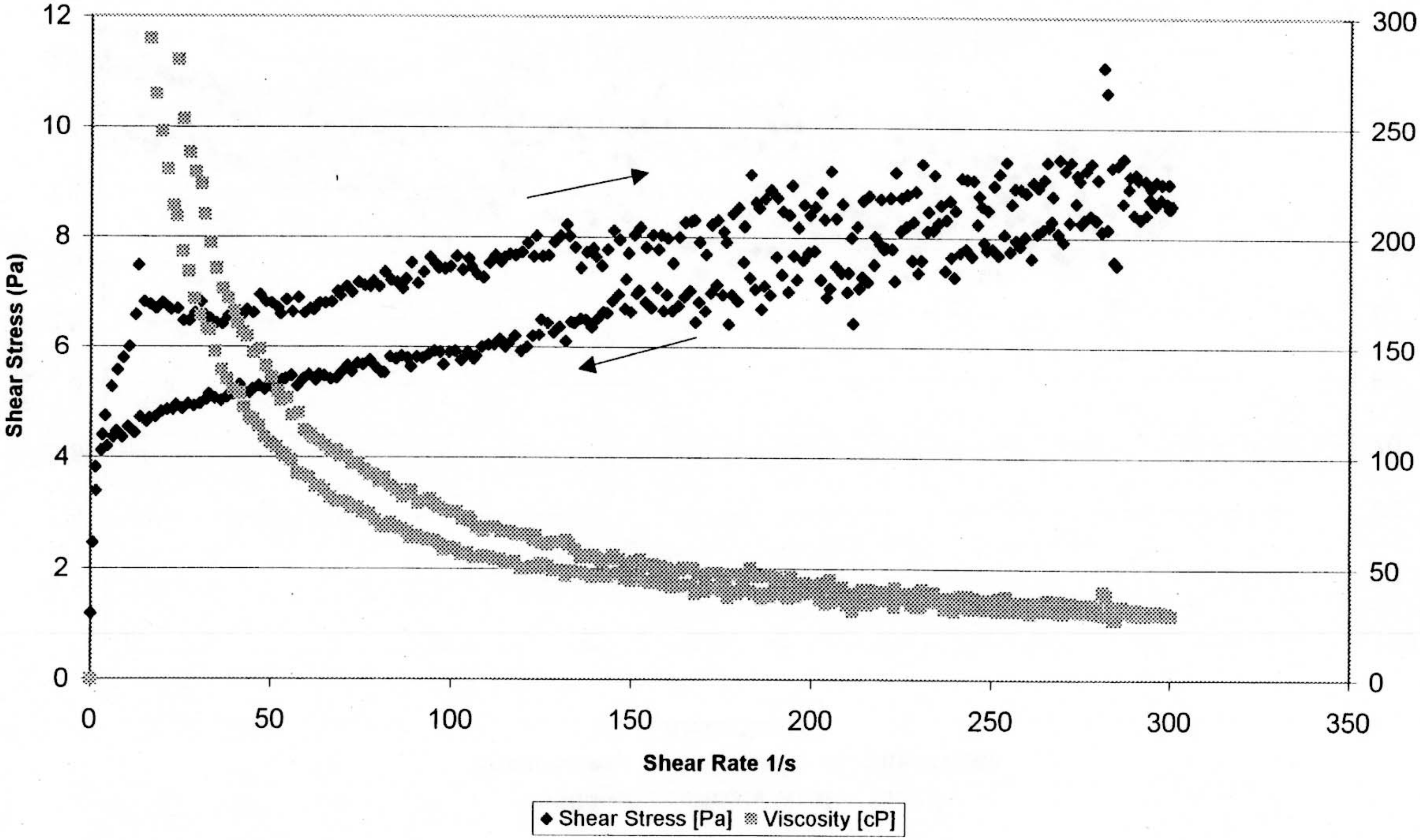
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste- 15wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



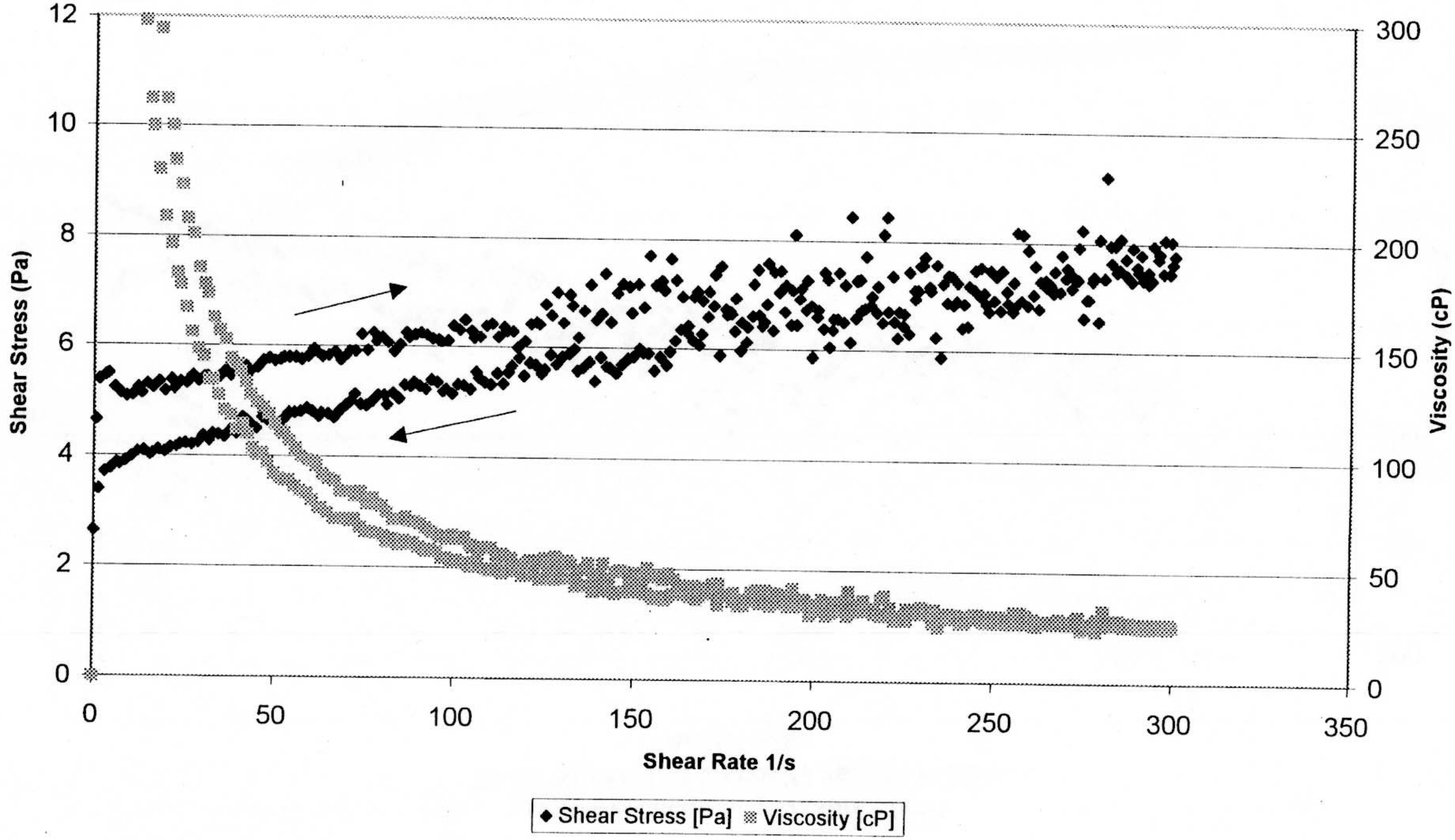
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste- 25wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #1

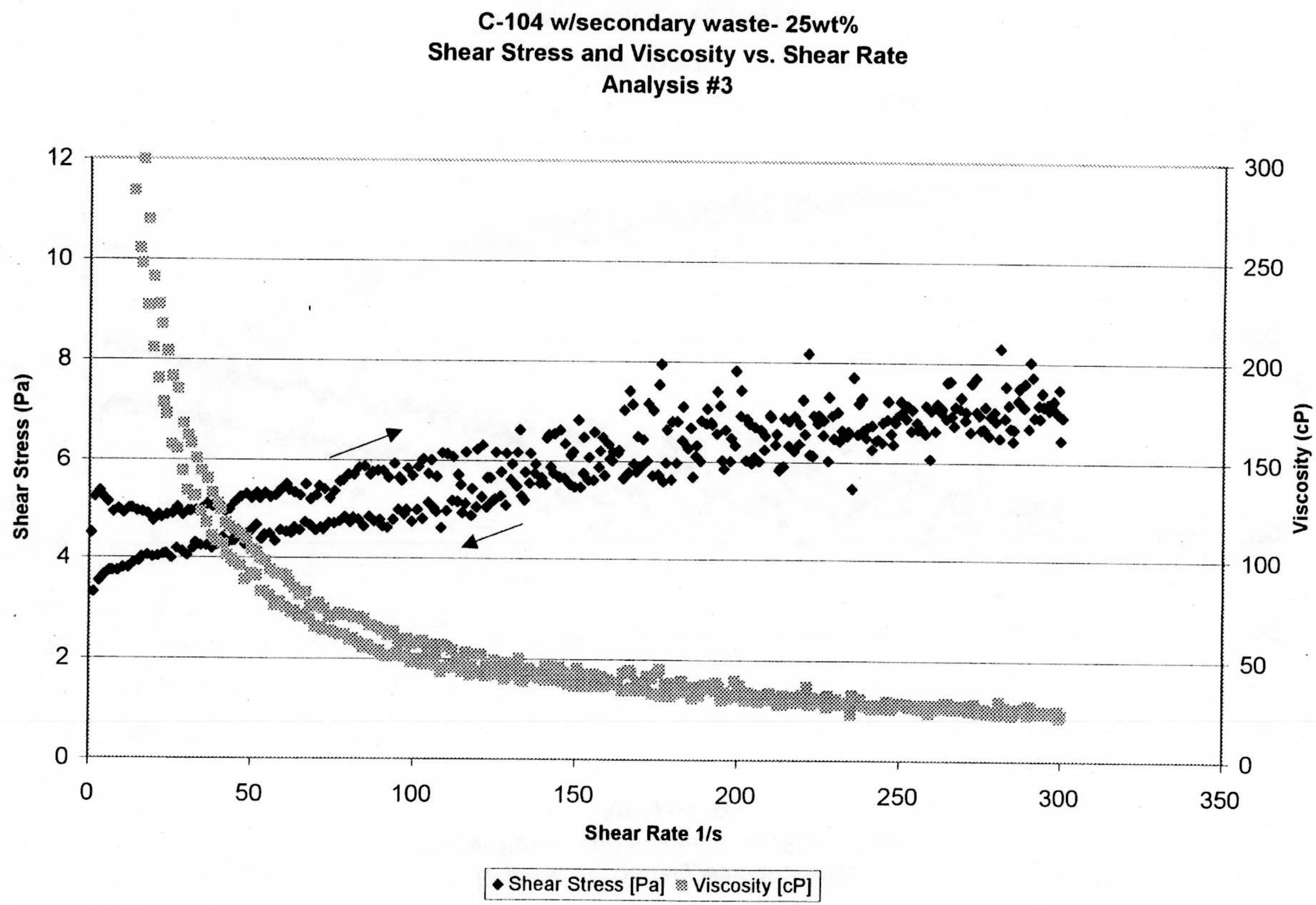


Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste- 25wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

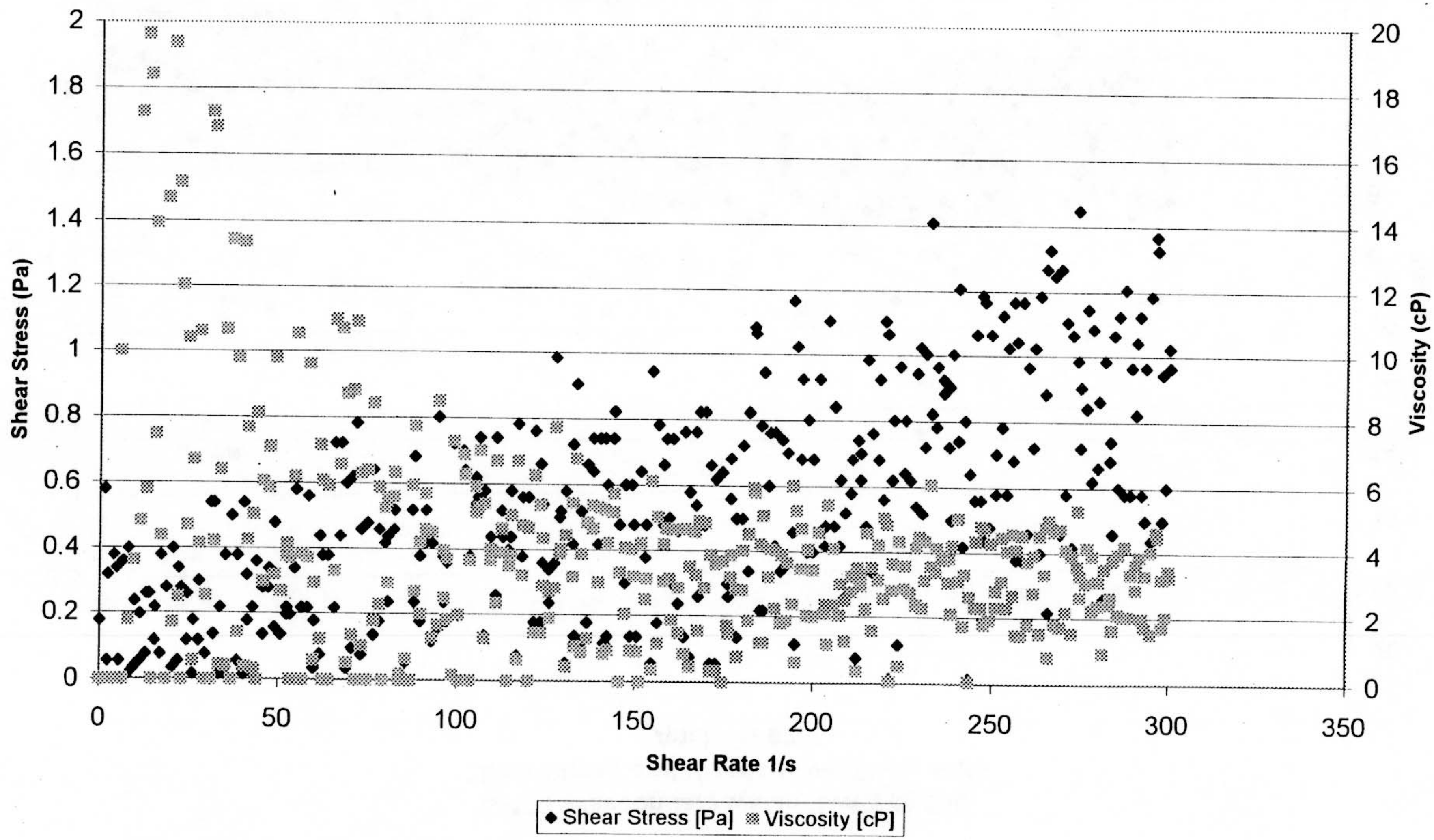


Measurement performed with a Haake M5 and SV I sensor



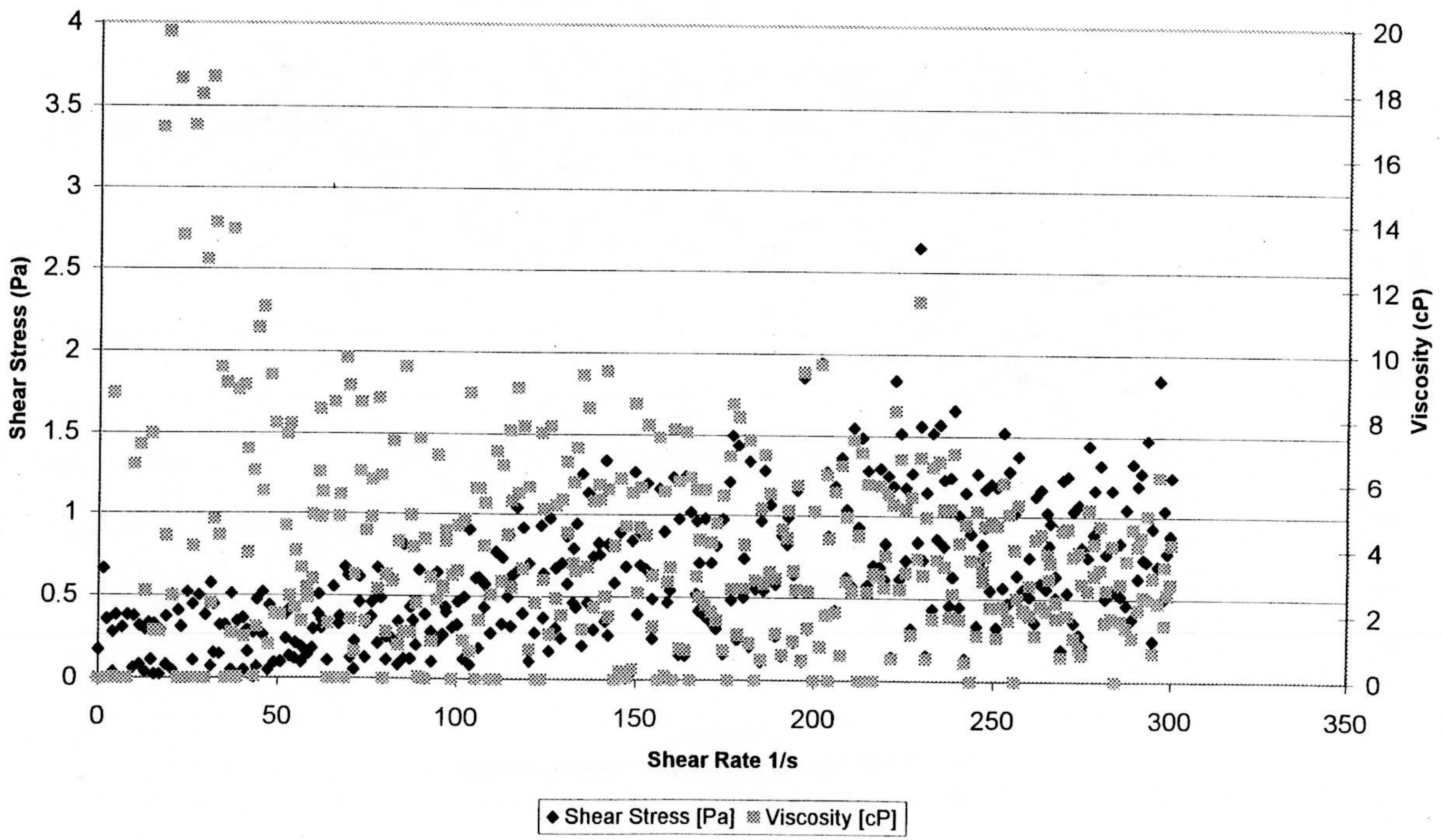
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste- 5wt% @ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #1

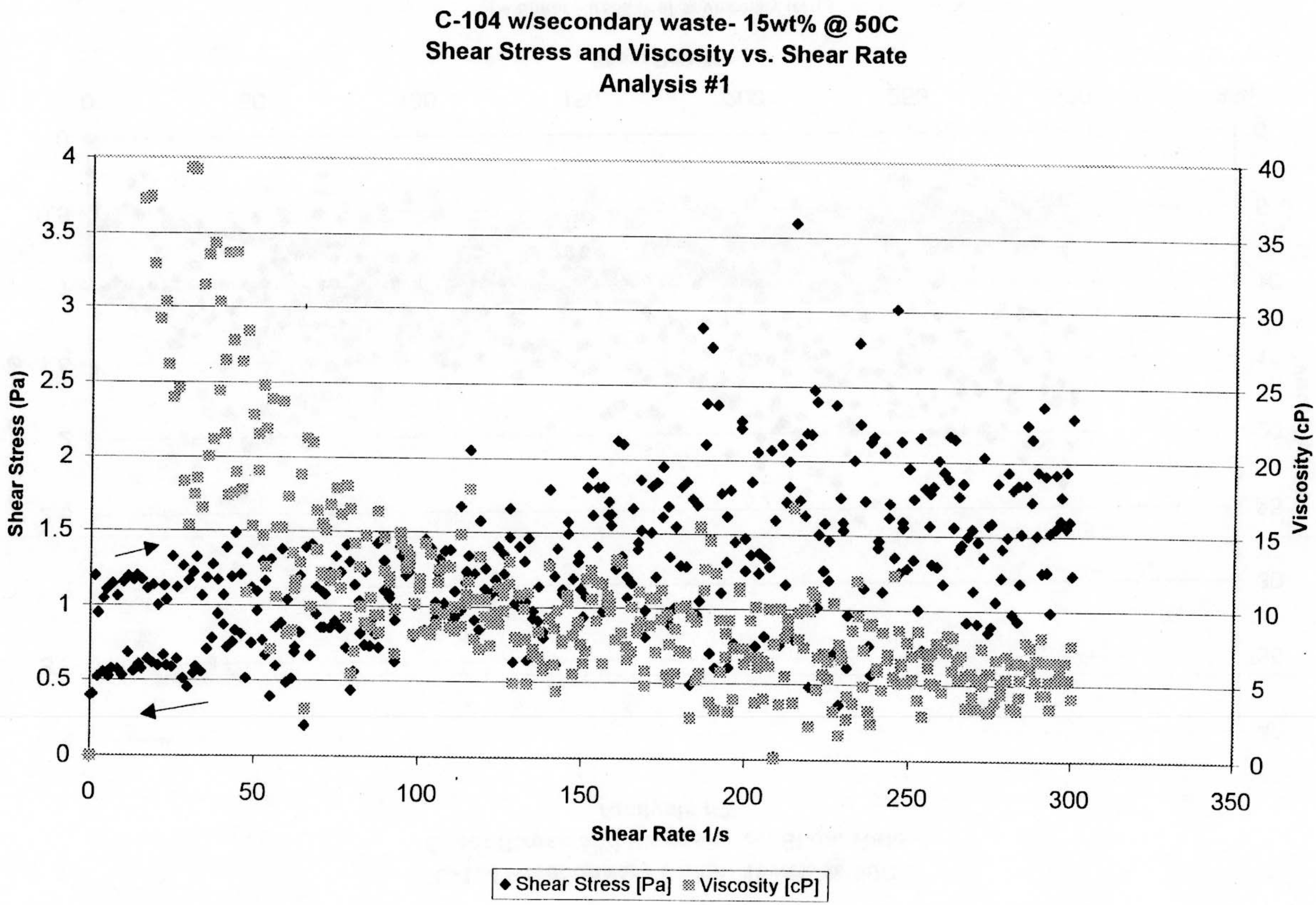


Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste- 5wt% @ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

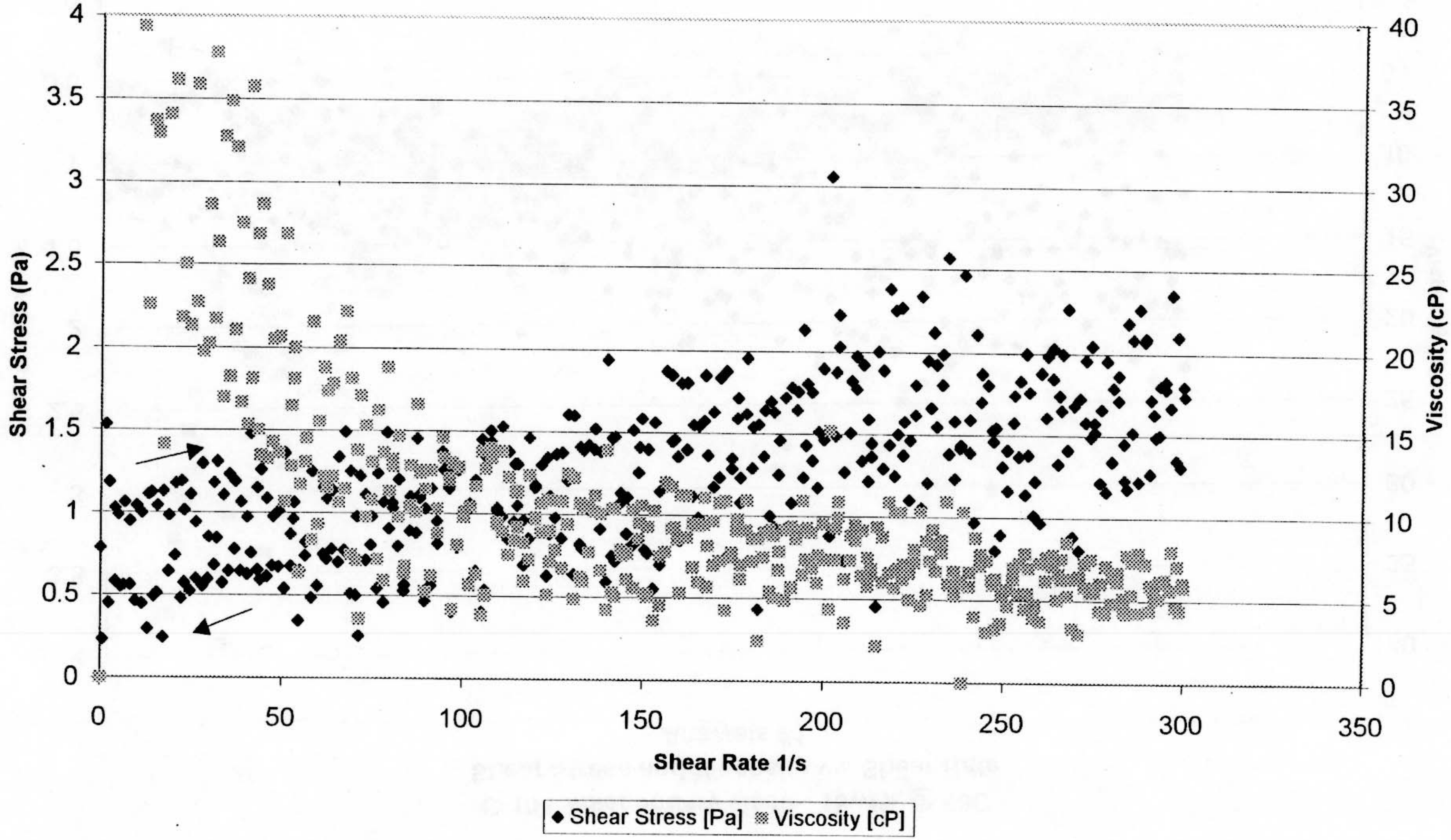


Measurement performed with a Haake M5 and SV I sensor



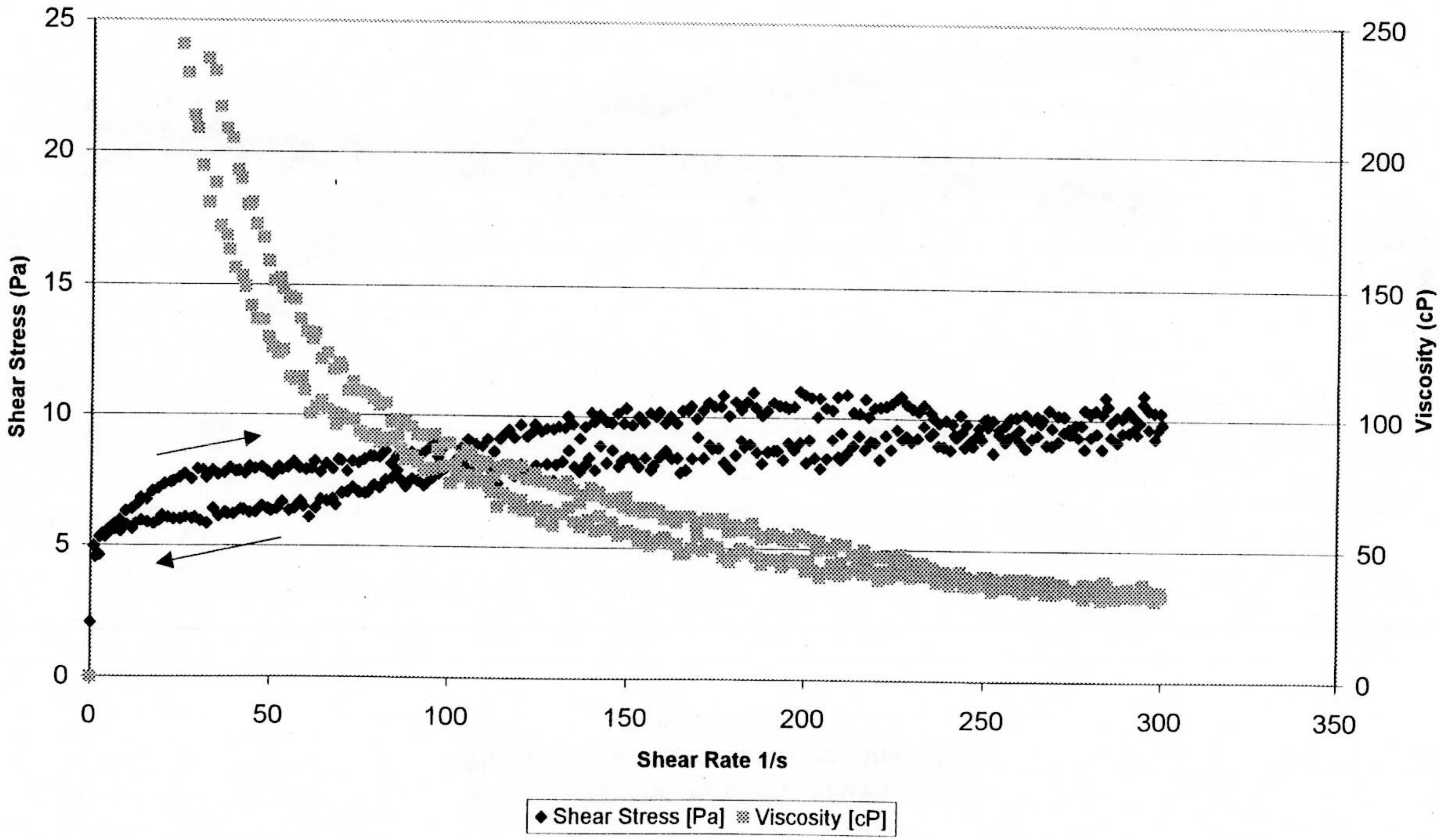
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste- 15wt% @ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



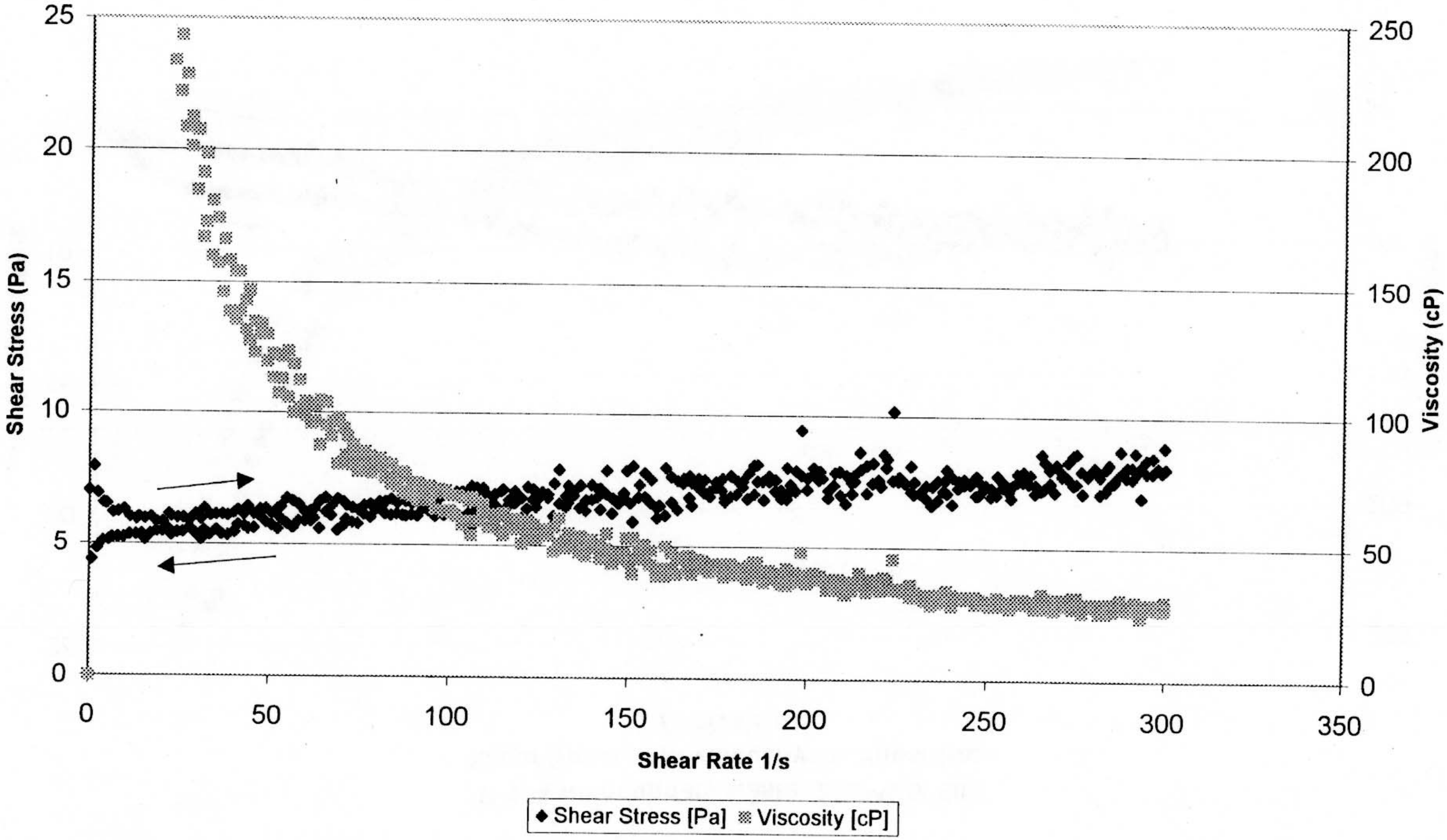
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste- 25wt% @ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #1



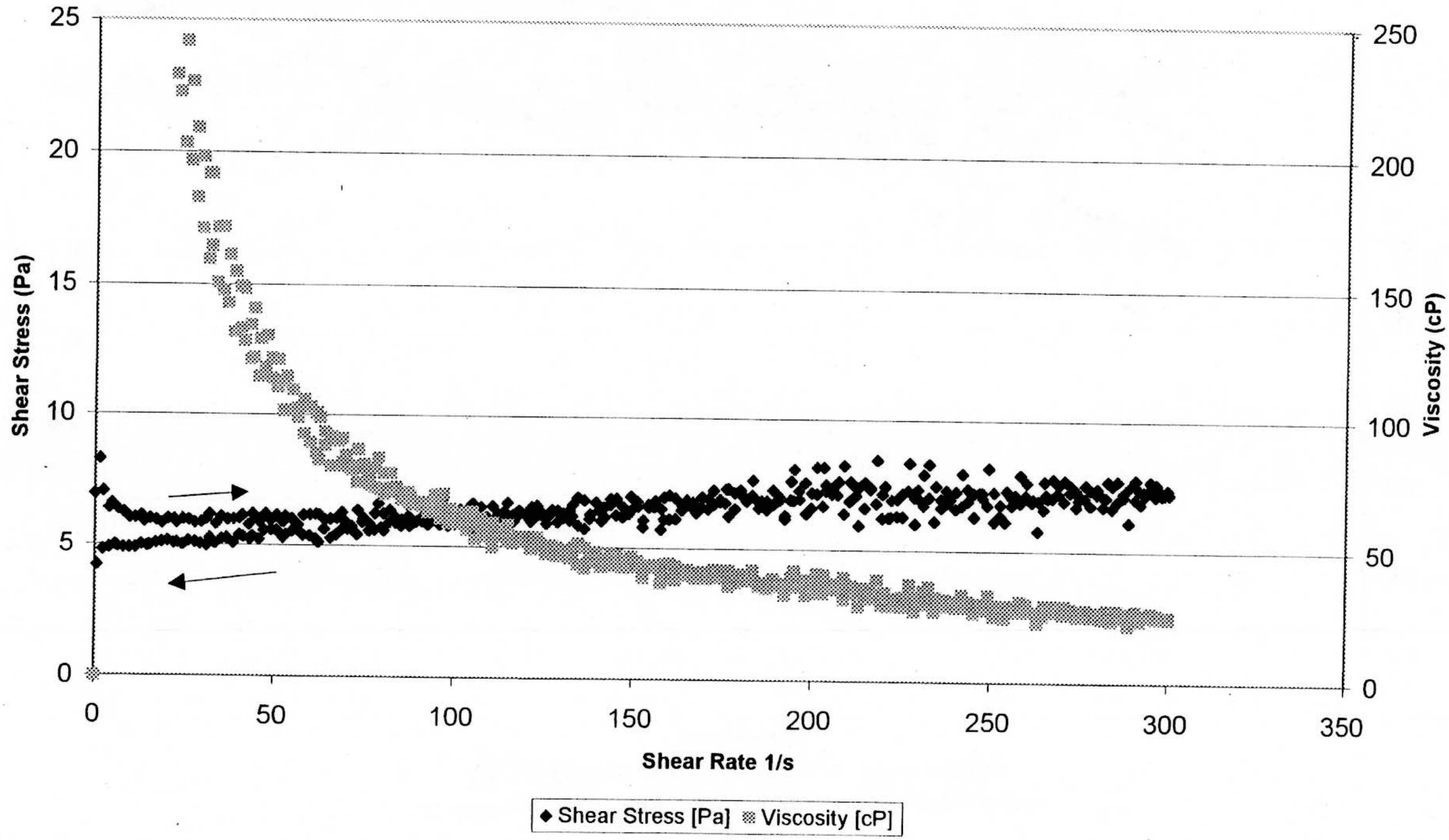
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste- 25wt% @ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



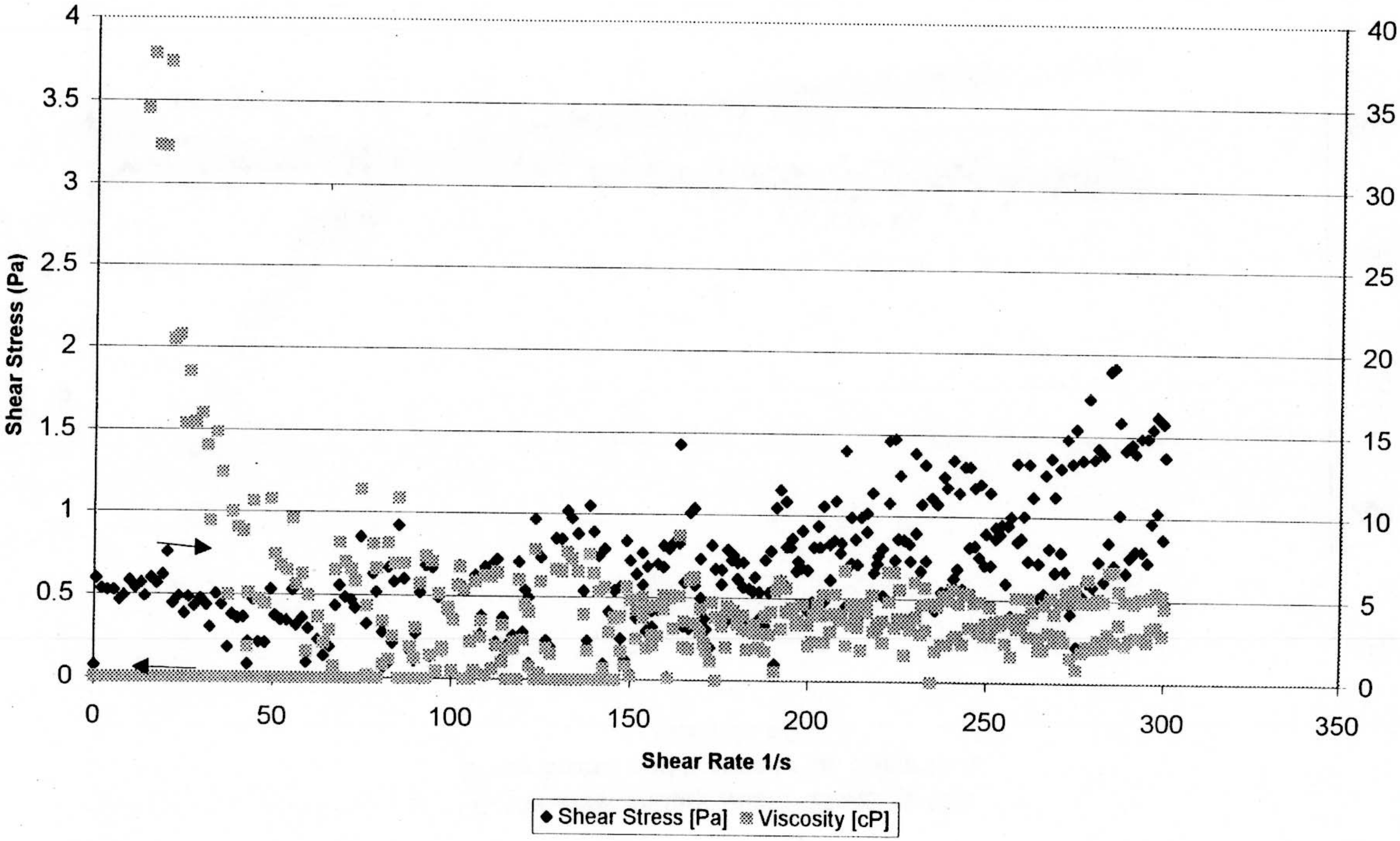
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste- 25wt% @ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #3

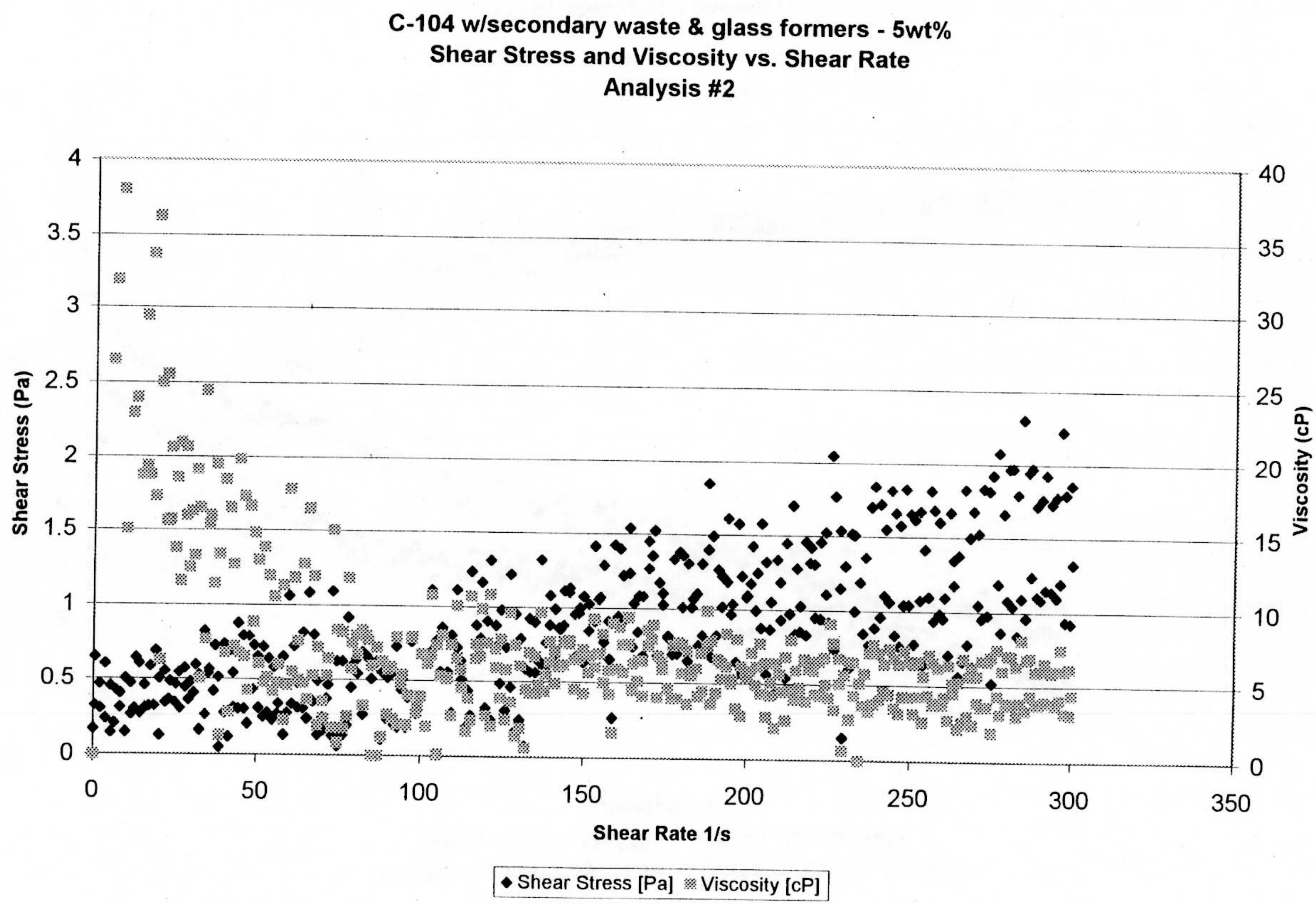


Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste & glass formers - 5wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #1

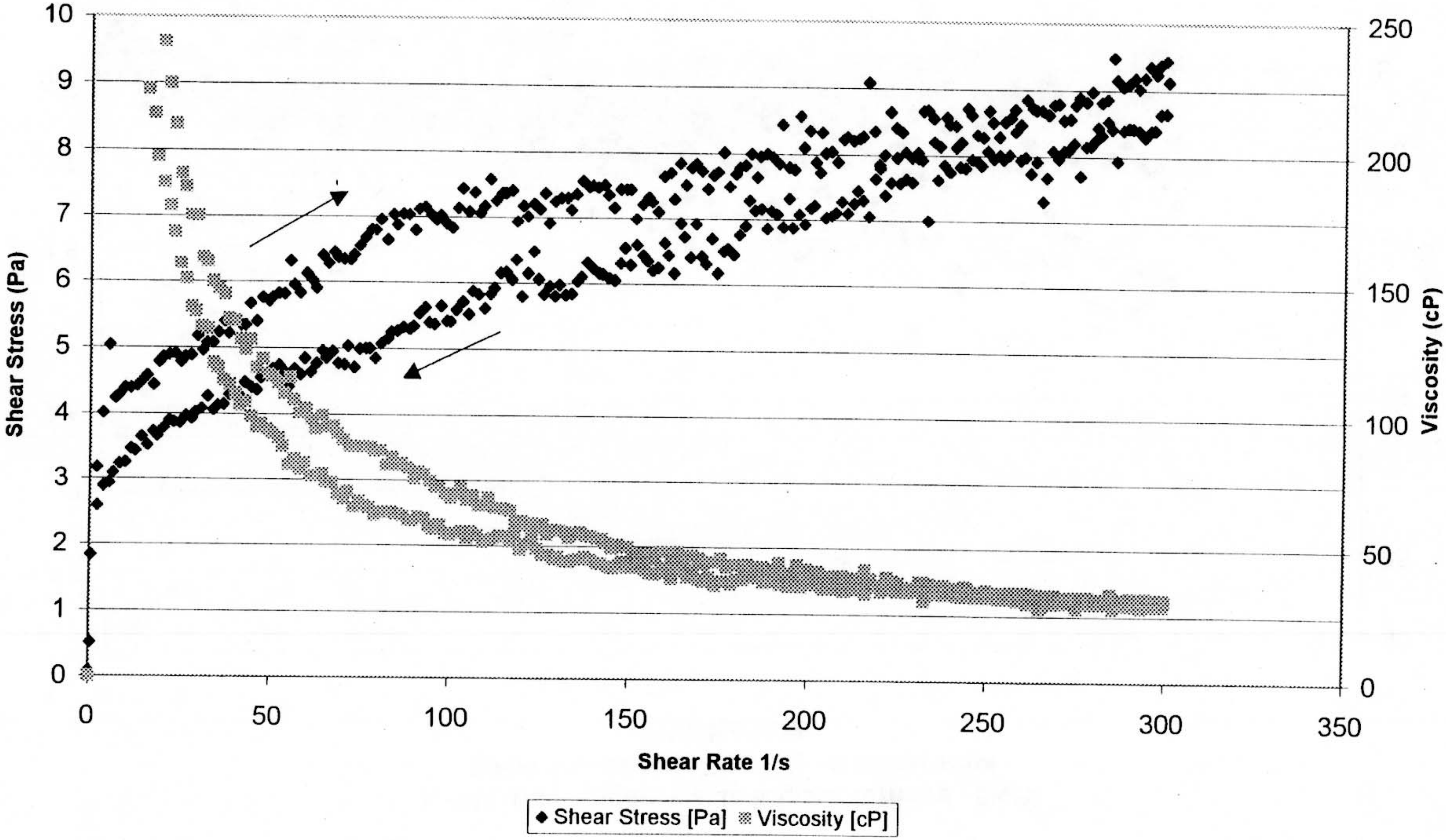


Measurement performed with a Haake M5 and SV I sensor



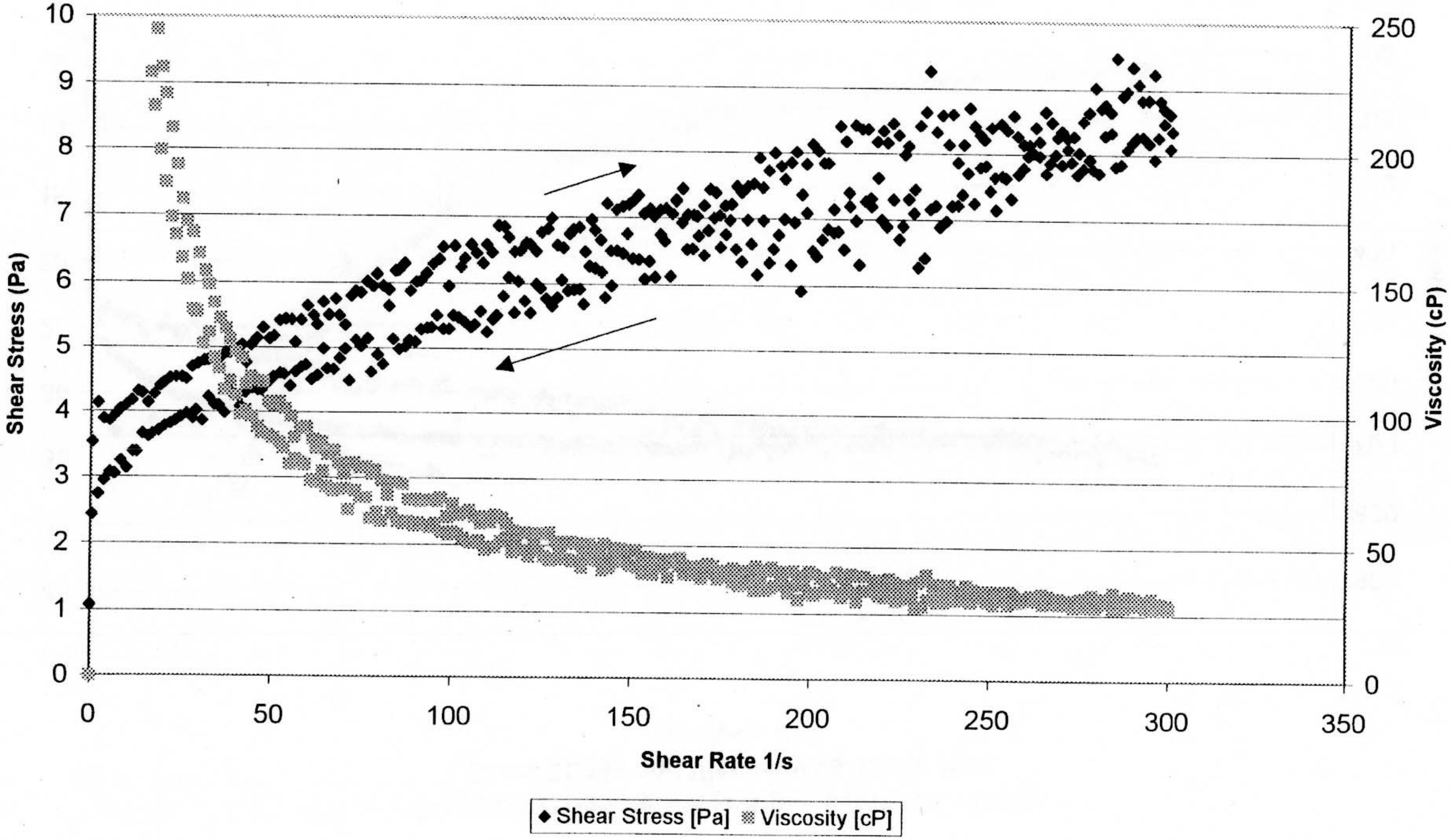
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste & glass formers - 15wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #1



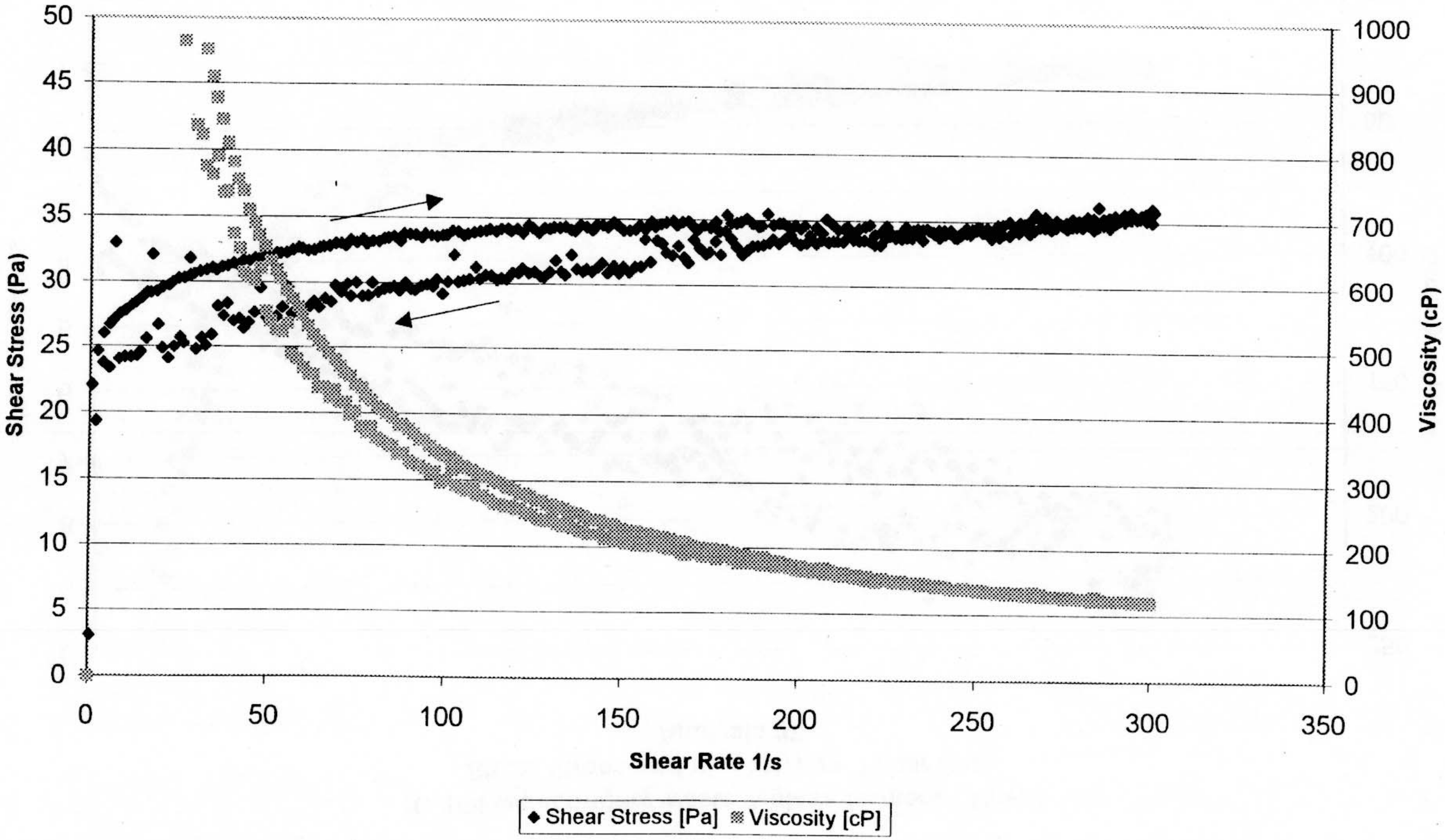
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste & glass formers - 15wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



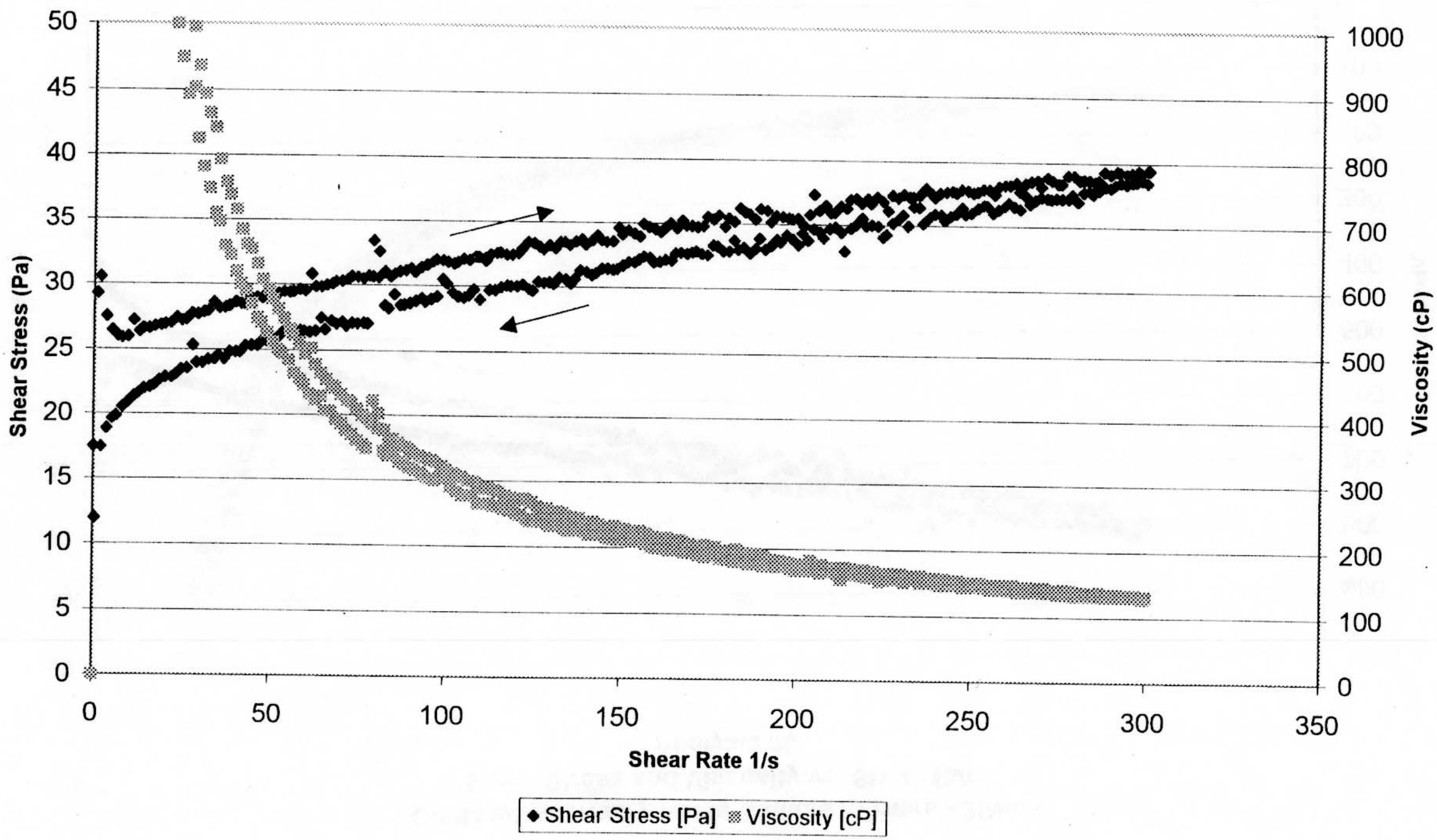
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste & glass formers - 25wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #1

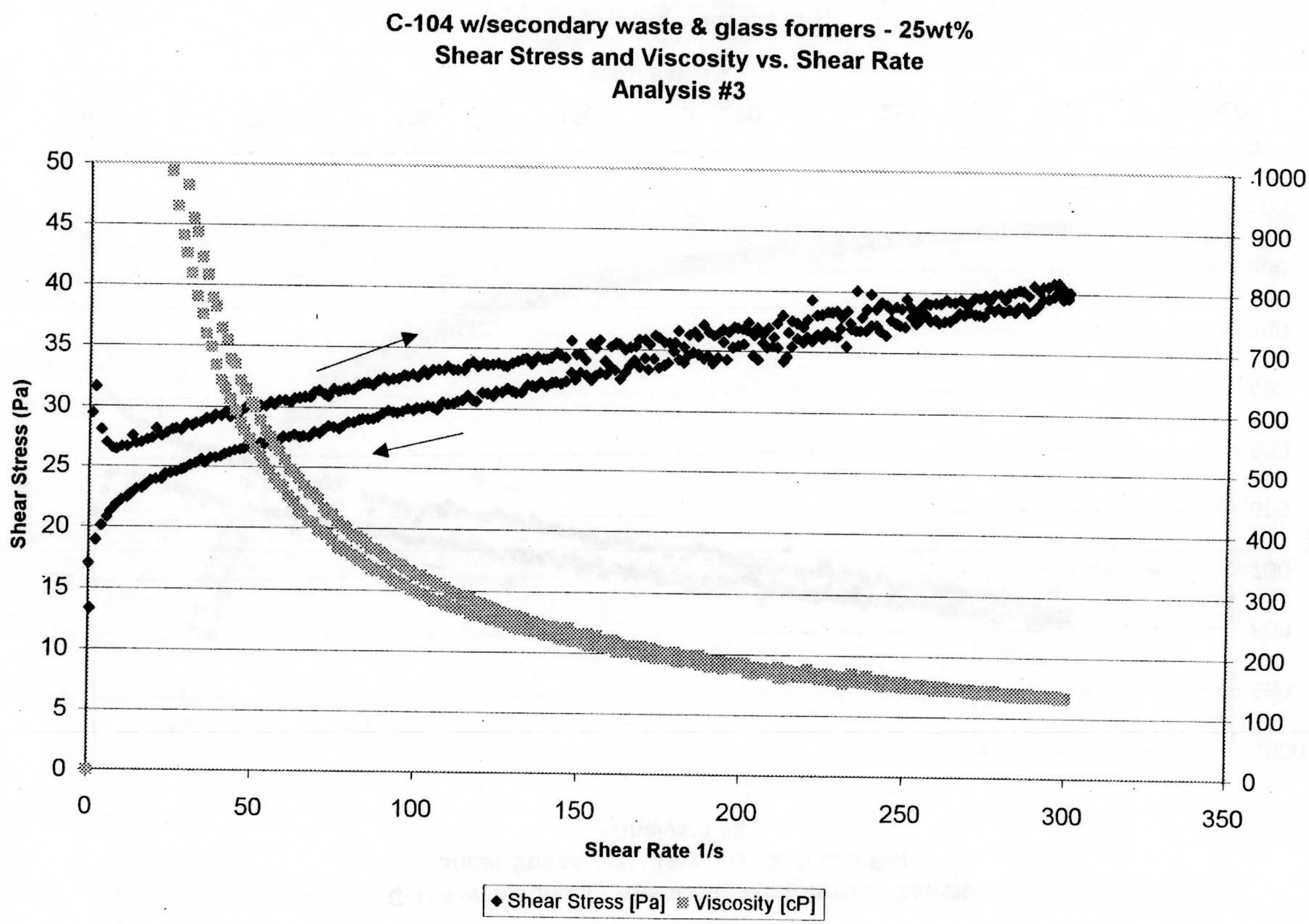


Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste & glass formers - 25wt%
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

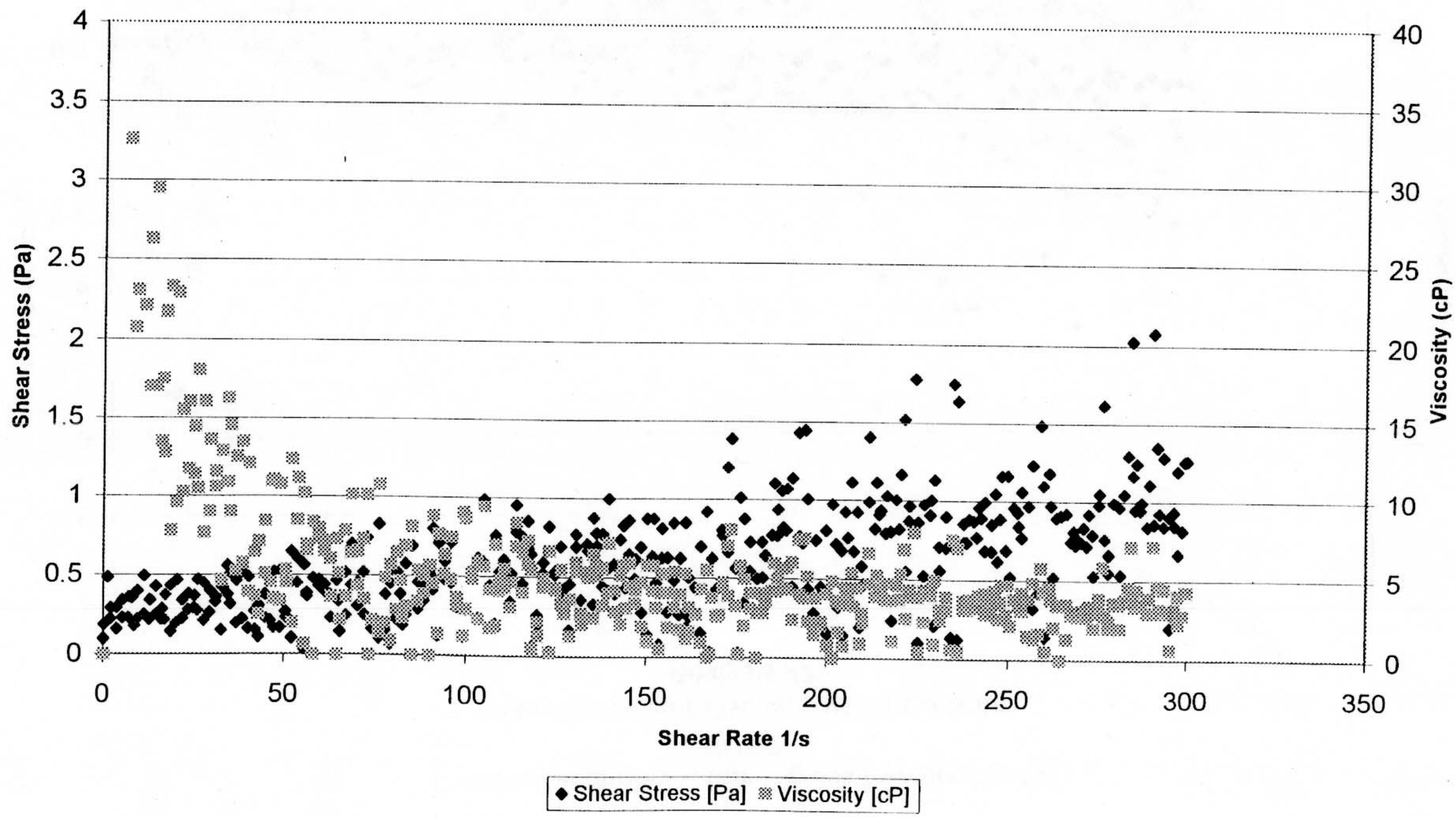


Measurement performed with a Haake M5 and SV I sensor



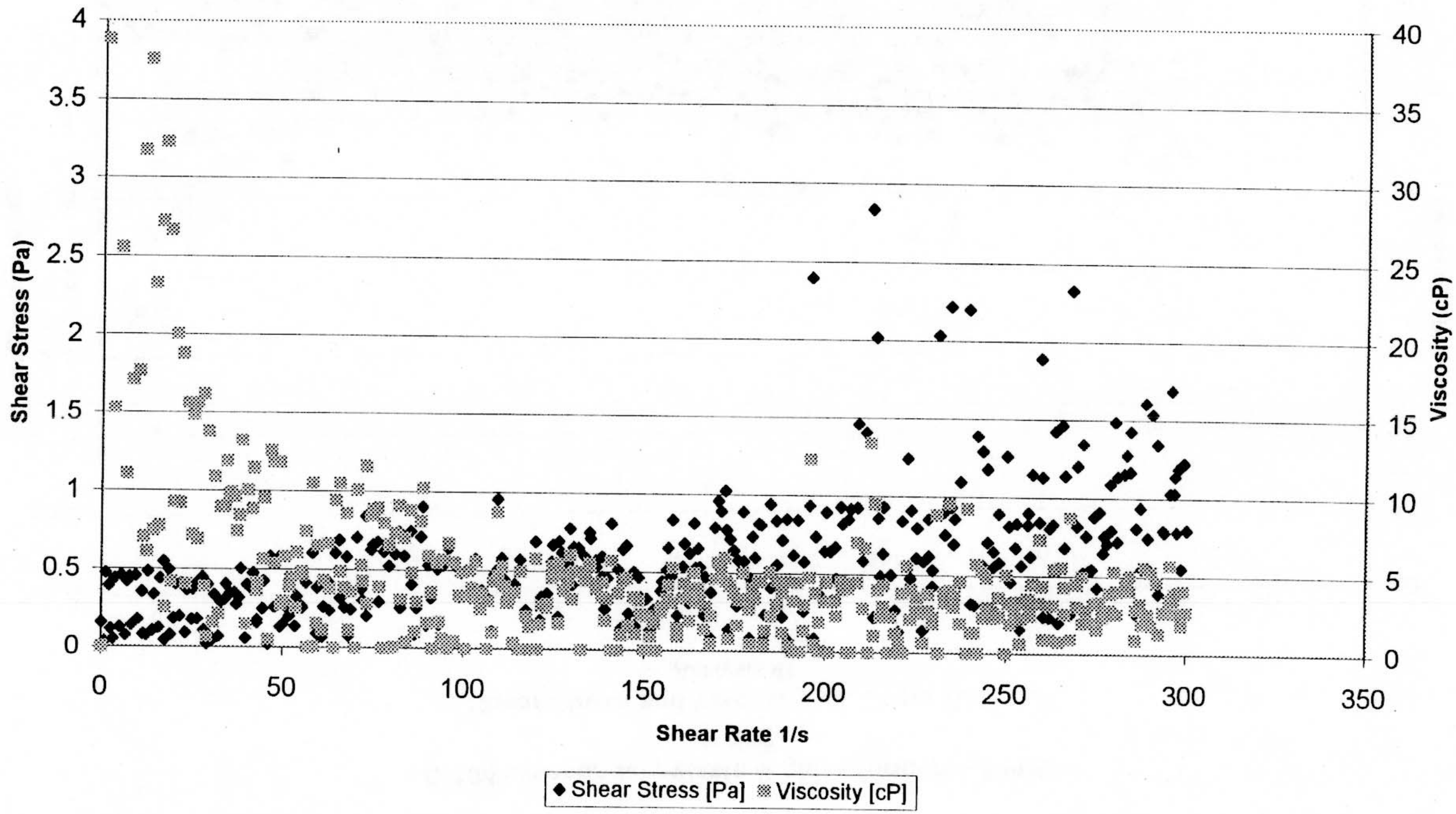
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste & glass formers - 5wt%
@ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #1



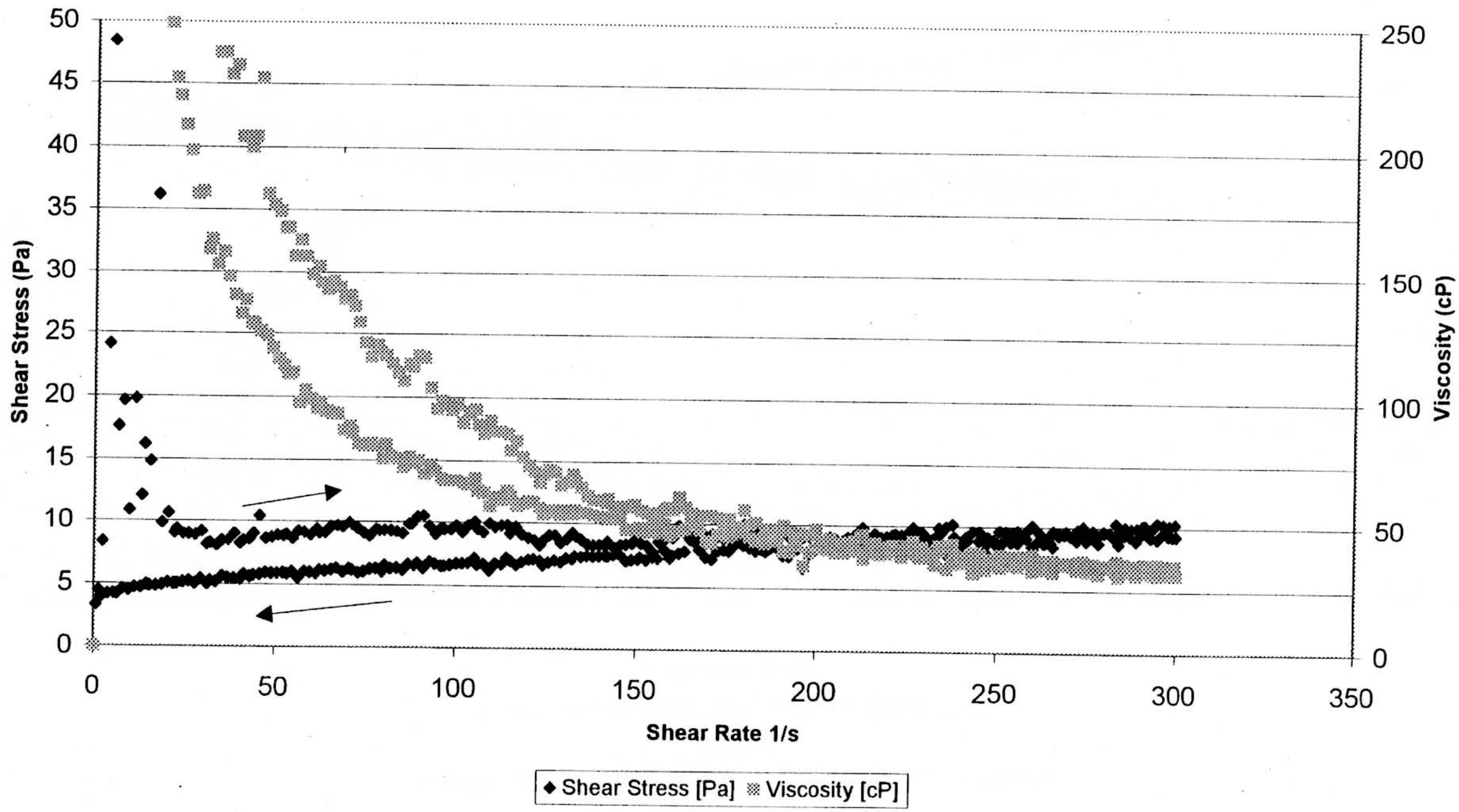
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste & glass formers - 5wt% @ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



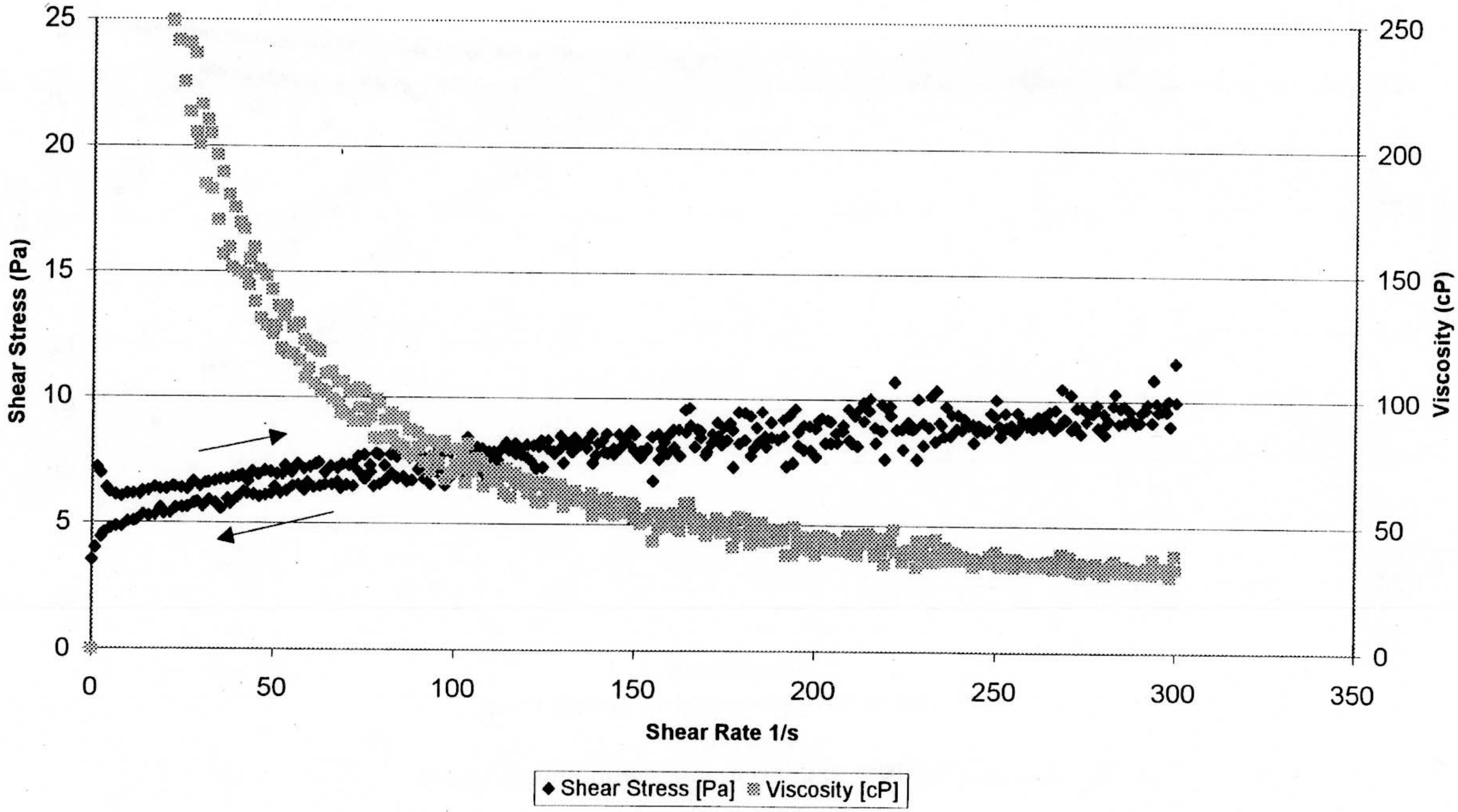
Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste & glass formers - 15wt% @ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #1

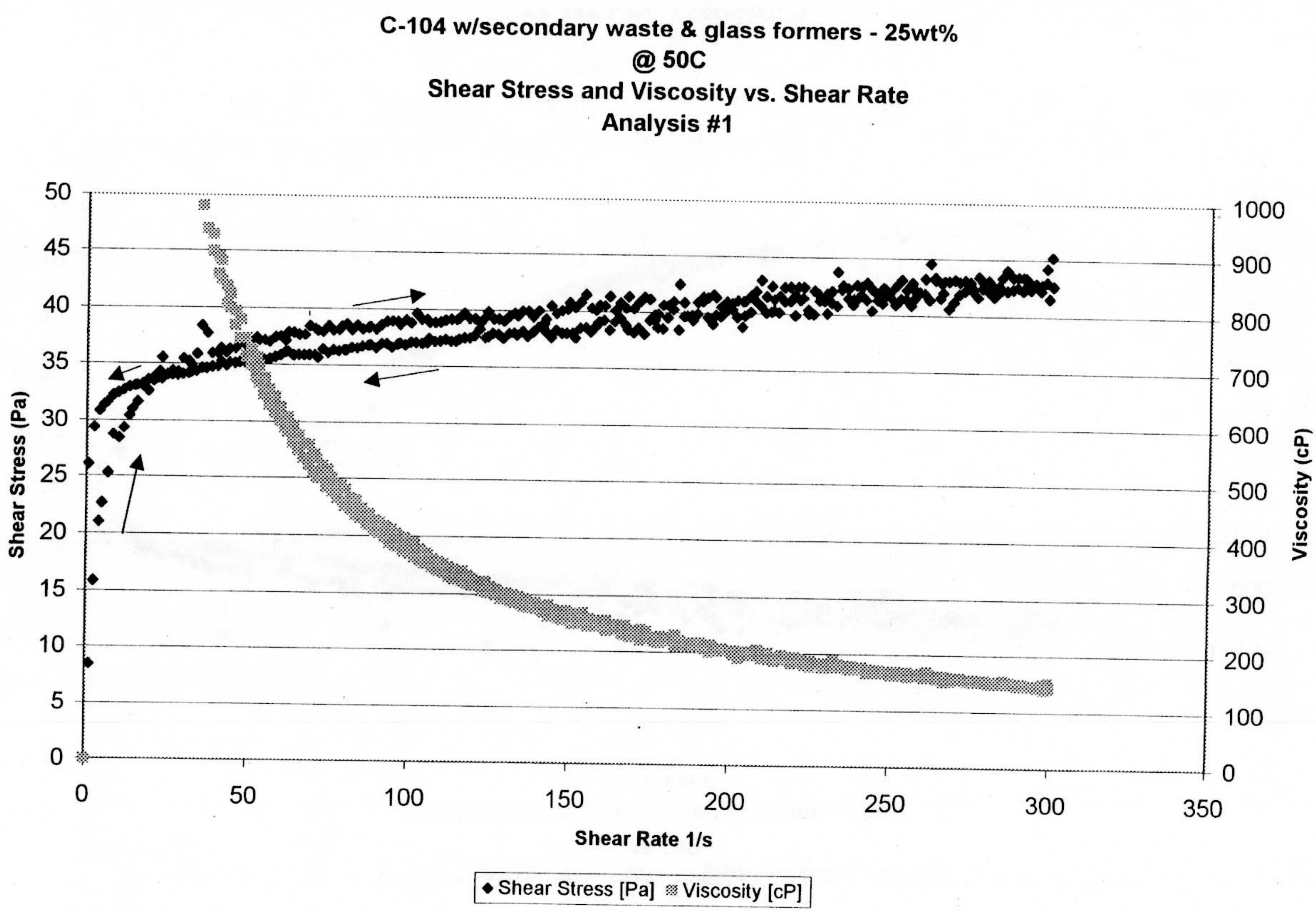


Measurement performed with a Haake M5 and SV I sensor

C-104 w/secondary waste & glass formers - 15wt%
@ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #2

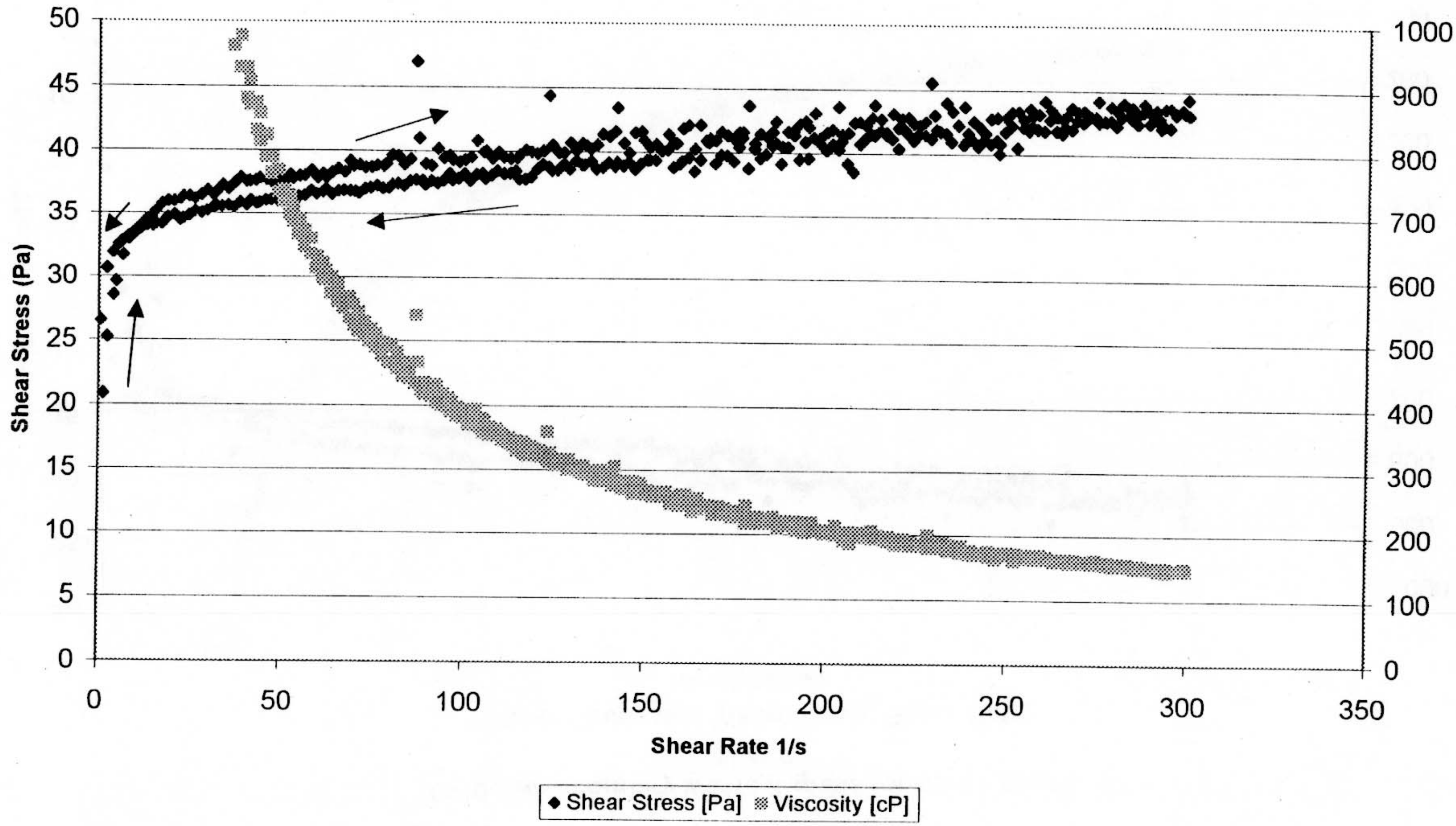


Measurement performed with a Haake M5 and SV I sensor

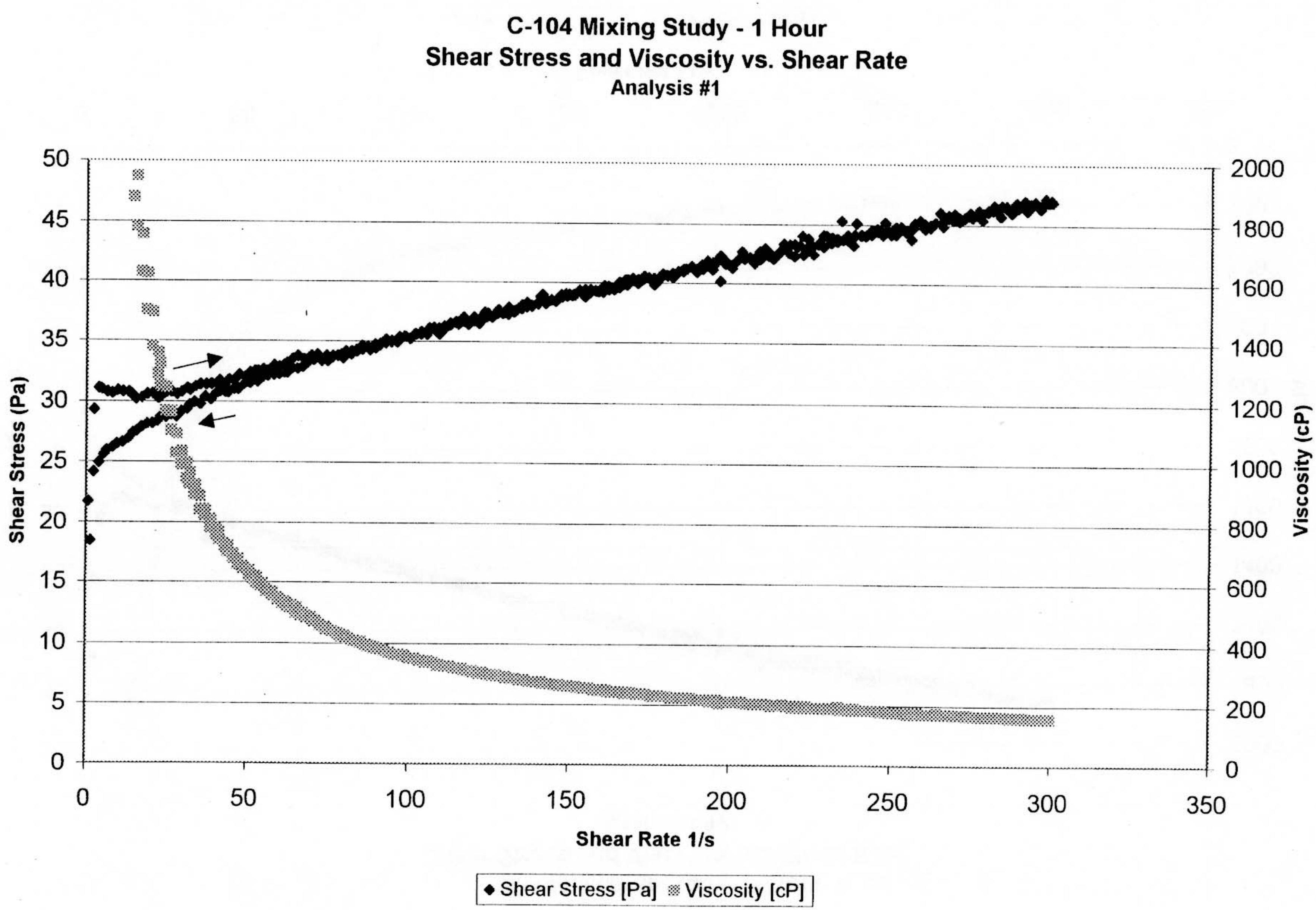


Measurement performed with a Haake M5 and SV I sensor

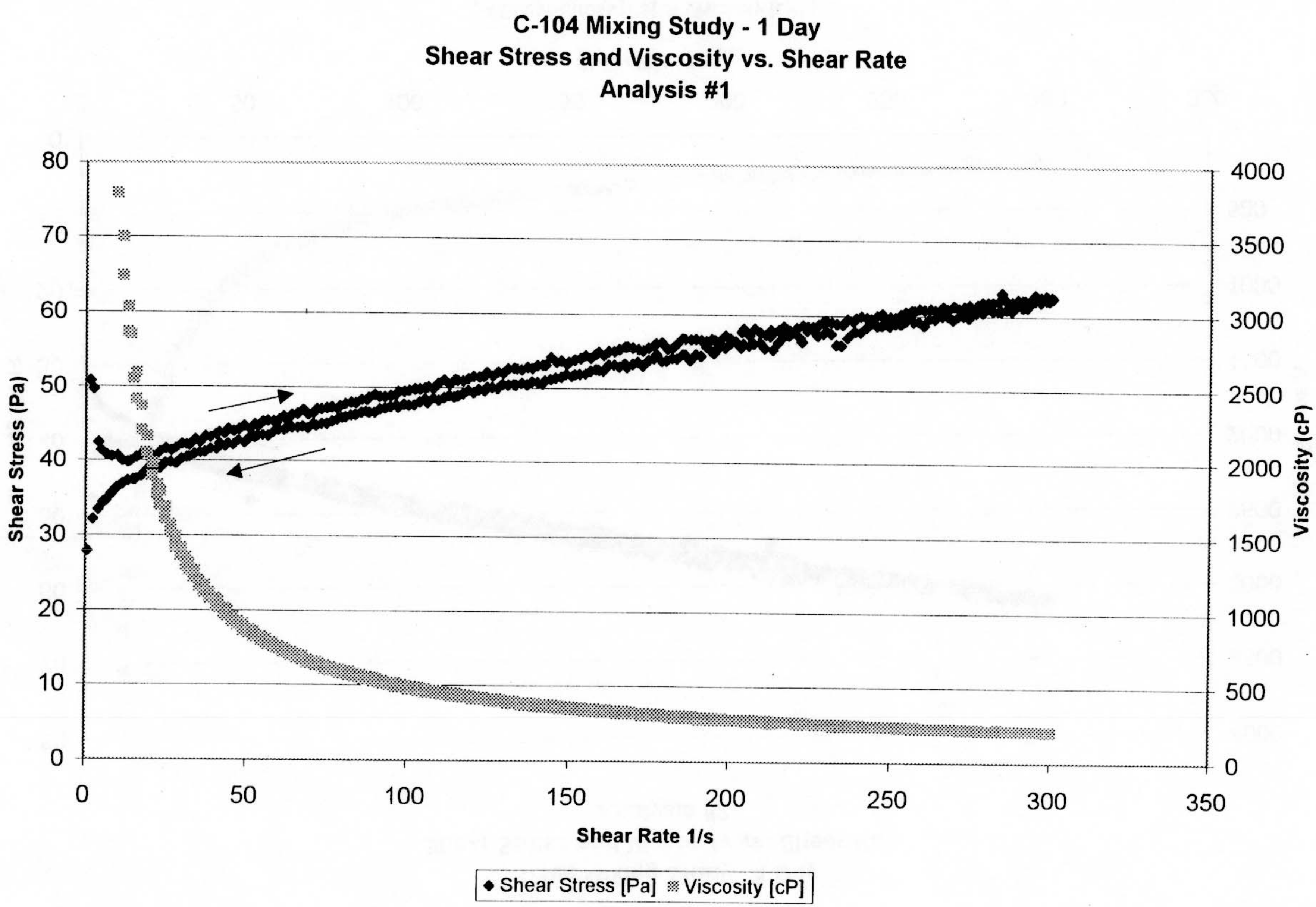
C-104 w/secondary waste & glass formers - 25wt%
@ 50C
Shear Stress and Viscosity vs. Shear Rate
Analysis #2



Measurement performed with a Haake M5 and SV I sensor

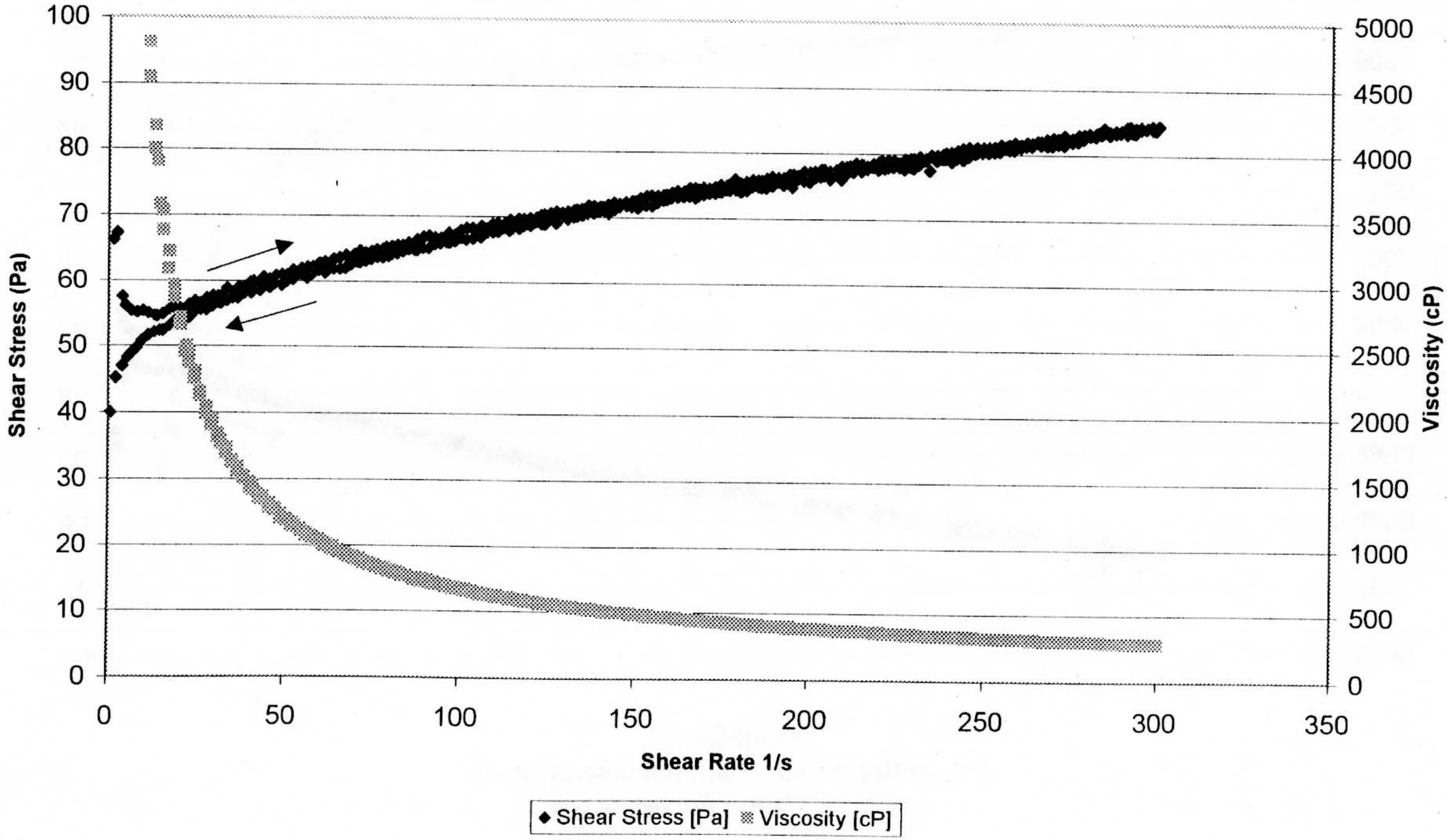


Measurement performed with a Haake M5 and SV I sensor



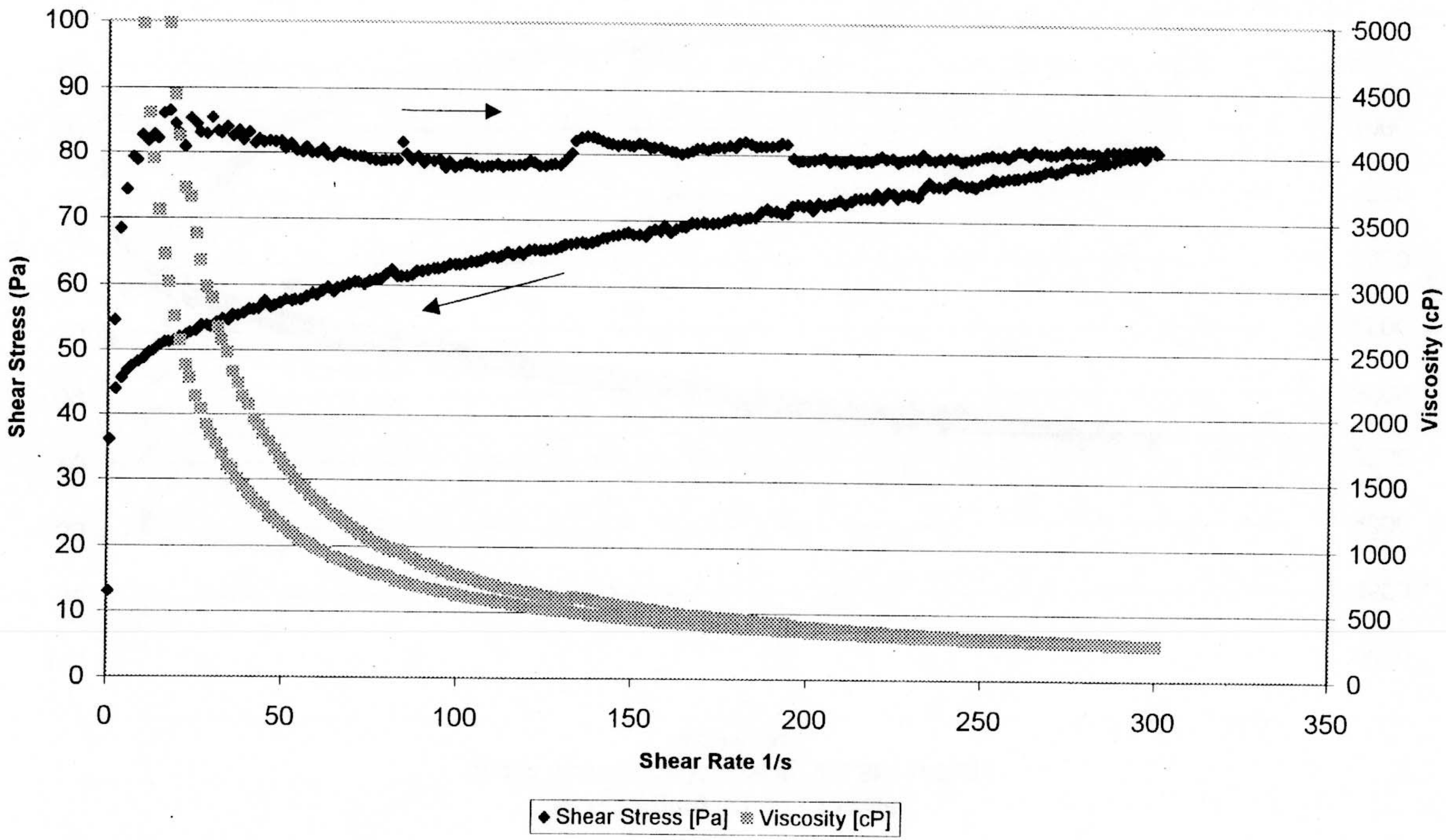
Measurement performed with a Haake M5 and SV I sensor

C-104 Mixing Study - 1 Week
Shear Stress and Viscosity vs. Shear Rate
Analysis #1



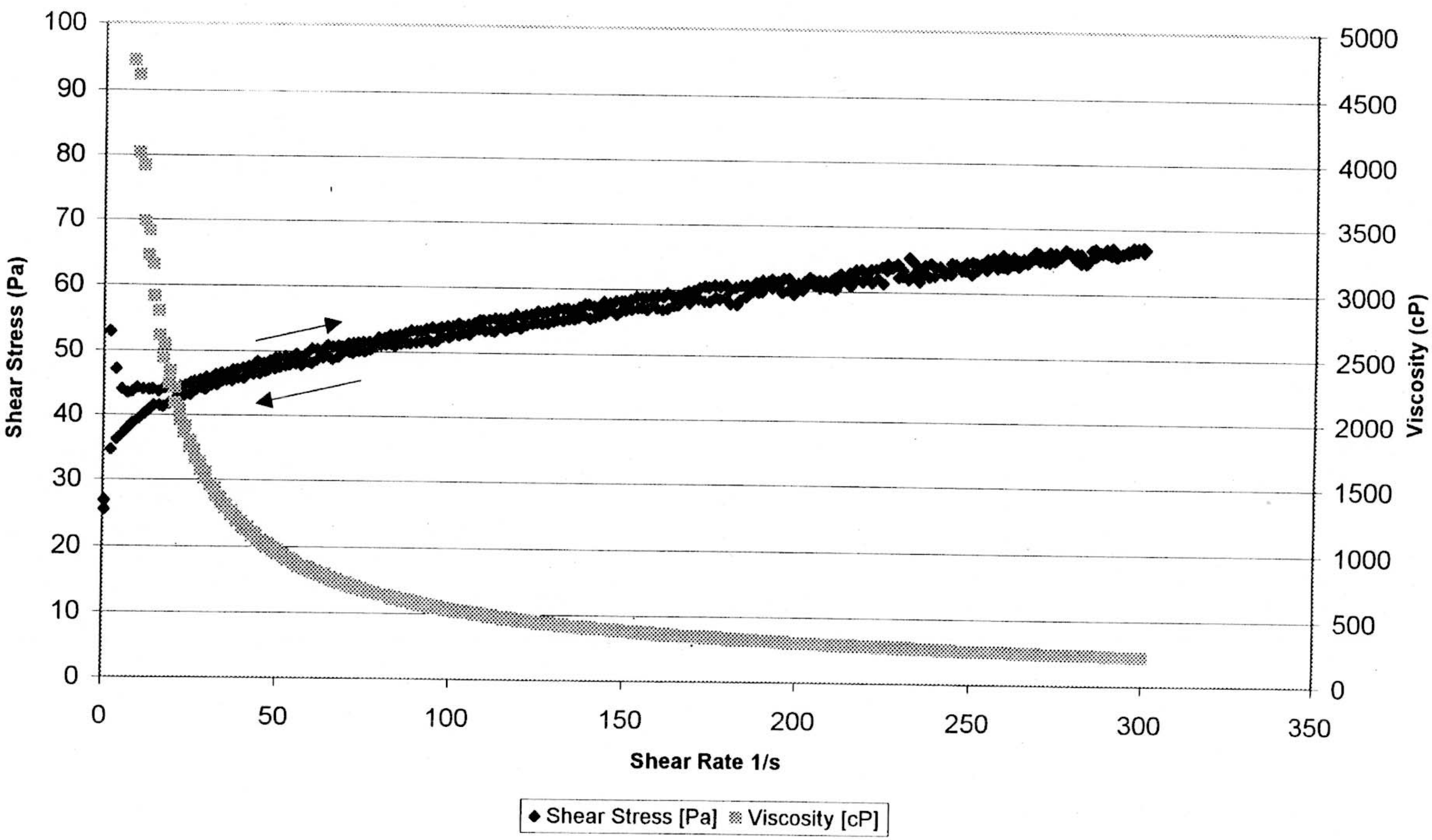
Measurement performed with a Haake M5 and SV I sensor

C-104 Aging Study - 1 Week
Shear Stress and Viscosity vs. Shear Rate
Analysis #1



Measurement performed with a Haake M5 and SV I sensor

C-104 Aging Study - 1 Week
Shear Stress and Viscosity vs. Shear Rate
Analysis #3



Measurement performed with a Haake M5 and SV I sensor

Average Viscosity Data

Viscosity in cP	C-104 Feed	25 C	25 C	25 C	25 C	C-104 Feed		50 C	50 C	50 C	50 C
File Name	Material	33 1/s	150 1/s	298 1/s	Other?	File Name	Material	33 1/s	150 1/s	298 1/s	Other?
050800B up (0-300 1/s)	C-104 5 wt% Feed	14.6	10.0	6.8		050800H up (0-300 1/s)	C-104 5 wt% Feed	4.6	5.2	5.0	
050800B down (300-0 1/s)	C-104 5 wt% Feed	5.9	2.8	5.8		050800H down (300-0 1/s)	C-104 5 wt% Feed	4.0	5.2	2.9	
050800C up (0-300 1/s)	C-104 5 wt% Feed - R	15.7	5.1	4.0		050800I up (0-300 1/s)	C-104 5 wt% Feed - R	6.4	5.3	2.4	
050800C down (300-0 1/s)	C-104 5 wt% Feed - R	3.2	2.9	2.1		050800I down (300-0 1/s)	C-104 5 wt% Feed - R	0.4	0.8	2.4	
Average all		9.8	5.2	4.7		Average all		3.8	4.1	3.2	
Average without 1st up curve		8.3	3.6	4.0		Average without 1st up curve		3.6	3.8	2.6	
050800D up (0-300 1/s)	C-104 15 wt% Feed	23.28	5.73	6.37		050800J up (0-300 1/s)	C-104 15 wt% Feed	14.5	3.7	5.0	
050800D down (300-0 1/s)	C-104 15 wt% Feed	10.33	5.705	5.347		050800J down (300-0 1/s)	C-104 15 wt% Feed			3.9	
050800E up (0-300 1/s)	C-104 15 wt% Feed - R	19.51	7.68	6.35		050800K up (0-300 1/s)	C-104 15 wt% Feed - R	16.5	6.6	6.2	
050800E down (300-0 1/s)	C-104 15 wt% Feed - R	8.358	5.54	4.87		050800K down (300-0 1/s)	C-104 15 wt% Feed - R	12.8	5.9	4.4	
Average all		15.4	6.2	5.7		Average all		14.6	5.4	4.9	
Average without 1st up curve		12.7	6.3	5.5		Average without 1st up curve		14.6	6.3	4.8	
050800F up (0-300 1/s)	C-104 25 wt% Feed	188.4	48.92	27.04		050800L up (0-300 1/s)	C-104 25 wt% Feed	245.1	57.8	28.9	
050800F down (300-0 1/s)	C-104 25 wt% Feed	160	44.62	26.06		050800L down (300-0 1/s)	C-104 25 wt% Feed	188.7	47.7	27.8	
050800G up (0-300 1/s)	C-104 25 wt% Feed - R	165.6	45.71	25.62		050800M up (0-300 1/s)	C-104 25 wt% Feed - R	173.8	47.9	25.2	
050800G down (300-0 1/s)	C-104 25 wt% Feed - R	145.6	42.8	24.9		050800M down (300-0 1/s)	C-104 25 wt% Feed - R	167.6	43.4	25.1	
Average all		164.9	45.5	25.9		Average all		193.8	49.2	26.7	
Average without 1st up curve		157.1	44.4	25.5		Average without 1st up curve		176.7	46.3	26.0	

Average Viscosity Data

Viscosity in cP	C-104 + Secondary waste + Glass Formers	25 C	25 C	25 C	25 C			50 C	50 C	50 C	50 C
File Name	Material	33 1/s	150 1/s	298 1/s	Other?	File Name	Material	33 1/s	150 1/s	298 1/s	Other?
062200A up (0-300 1/s)	C-104 5 wt% Feed	10.0	4.7	5.4		062300B up (0-300 1/s)	C-104 5 wt% Feed	13.2	4.9	4.1	
062200A down (300-0 1/s)	C-104 5 wt% Feed	0.0	0.5	2.6		062300B down (300-0 1/s)	C-104 5 wt% Feed	8.0	1.0	2.4	
062200B up (0-300 1/s)	C-104 5 wt% Feed - R	18.3	7.2	6.1		062300C up (0-300 1/s)	C-104 5 wt% Feed - R	9.1	2.1	4.0	
062200B down (300-0 1/s)	C-104 5 wt% Feed - R	4.6	4.3	2.7		062300C down (300-0 1/s)	C-104 5 wt% Feed - R	5.8	0.3	2.0	
Average all		8.2	4.2	4.2		Average all		9.0	2.0	3.1	
Average without 1st up curve		7.6	4.0	3.8		Average without 1st up curve		7.6	1.1	2.8	
062200C up (0-300 1/s)	C-104 15 wt% Feed	154.7	49.5	31.5		062300D up (0-300 1/s)	C-104 15 wt% Feed	248.5	57.6	34.2	
062200C down (300-0 1/s)	C-104 15 wt% Feed	125.9	42.01	28.4		062300Ddown (300-0 1/s)	C-104 15 wt% Feed	155.3	47.1	31.4	
062200D up (0-300 1/s)	C-104 15 wt% Feed - R	145.7	48.3	29.2		062300E up (0-300 1/s)	C-104 15 wt% Feed - R	200.1	51.5	34.3	
062200D down (300-0 1/s)	C-104 15 wt% Feed - R	126.7	42.6	27.65		062300E down (300-0 1/s)	C-104 15 wt% Feed - R	177.5	55.6	30.5	
Average all		138.3	45.6	29.2		Average all		195.4	53.0	32.6	
Average without 1st up curve		132.8	44.3	28.4		Average without 1st up curve		177.6	51.4	32.1	
062200F up (0-300 1/s)	C-104 25 wt% Feed	761.1	208.8	120		062300G up (0-300 1/s)	C-104 25 wt% Feed	1064	272.6	148.3	
062200F down (300-0 1/s)	C-104 25 wt% Feed	938.6	227.2	117		062300G down (300-0 1/s)	C-104 25 wt% Feed	1041	254.4	144	
062200H up (0-300 1/s)	C-104 25 wt% Feed - R	869.4	230.4	135.5		062300J up (0-300 1/s)	C-104 25 wt% Feed - R	1121.0	271.4	145.6	
062200H down (300-0 1/s)	C-104 25 wt% Feed - R	771.7	220.4	132		062300J down (300-0 1/s)	C-104 25 wt% Feed - R	1077.0	262.0	146.0	
Average all		835.2	221.7	126.1		Average all		1075.8	265.1	146.0	
Average without 1st up curve		859.9	226.0	128.2		Average without 1st up curve		1079.7	262.6	145.2	

<u>Average Viscosity Data</u>

Viscosity in cP	**AZ-102 Feed**	25 C	25 C	25 C	25 C			50 C	50 C	50 C	50 C
File Name	Material	33 1/s	150 1/s	298 1/s	Other?	File Name	Material	33 1/s	150 1/s	298 1/s	Other?
051200A up (0-300 1/s)	AZ-102 5 wt% Feed	13.4	6.0	5.3		051200H up (0-300 1/s)	AZ-102 5 wt% Feed	18.7	5.2	4.8	
051200A down (300-0 1/s)	AZ-102 5 wt% Feed	8.0	2.5	4.4		051200H down (300-0 1/s)	AZ-102 5 wt% Feed	5.1	4.1	4.4	
051200B up (0-300 1/s)	AZ-102 5 wt% Feed - R	15.7	8.5	5.7		051200I up (0-300 1/s)	AZ-102 5 wt% Feed - R	12.4	4.7	4.8	
051200B down (300-0 1/s)	AZ-102 5 wt% Feed - R	11.3	5.9	5.2		051200I down (300-0 1/s)	AZ-102 5 wt% Feed - R	10.7	4.9	3.3	
Average all		12.1	5.7	5.2		Average all		11.7	4.7	4.3	
Average without 1st up curve		11.7	5.6	5.1		Average without 1st up curve		9.4	4.6	4.2	
051200C up (0-300 1/s)	AZ-102 15 wt% Feed	527.9	153.1	79.1		051200J up (0-300 1/s)	AZ-102 15 wt% Feed	529.1	150.9	85.7	
051200C down (300-0 1/s)	AZ-102 15 wt% Feed	537.9	142.7	78.5		051200J down (300-0 1/s)	AZ-102 15 wt% Feed	626.3	159.0	85.7	
051200D up (0-300 1/s)	AZ-102 15 wt% Feed - R	517.1	141.3	78.99		051200K up (0-300 1/s)	AZ-102 15 wt% Feed - R	523.1	139.5	76.1	
051200D down (300-0 1/s)	AZ-102 15 wt% Feed - R	517.7	139.1	78.36		051200K down (300-0 1/s)	AZ-102 15 wt% Feed - R	546.1	143.6	77.7	
Average all		525.2	144.1	78.7		Average all		556.2	148.3	81.3	
Average without 1st up curve		524.2	141.0	78.6		Average without 1st up curve		565.2	147.4	79.8	
060800C up (0-300 1/s)	AZ-102 20 wt% Feed	917.1	234.6	129.8							
060800C down (300-0 1/s)	AZ-102 20 wt% Feed	890.5	231.5	127.4							
060800D up (0-300 1/s)	AZ-102 20 wt% Feed - R										
060800D down (300-0 1/s)	AZ-102 20 wt% Feed - R										
Average all		903.8	233.1	128.6							
Average without 1st up curve		890.5	231.5	127.4							
051200E up (0-300 1/s)	AZ-102 25 wt% Feed	6220	1493	802.5							
051200E down (300-0 1/s)	AZ-102 25 wt% Feed	5281	1446	799							
051200F up (0-300 1/s)	AZ-102 25 wt% Feed - R	4047	1167	656.7							
051200F down (300-0 1/s)	AZ-102 25 wt% Feed - R	4002	1162	655.1							
051200G up (0-300 1/s)	AZ-102 25 wt% Feed - R	4017	1157	651.5							
051200G down (300-0 1/s)	AZ-102 25 wt% Feed - R	4016	1156	650.7							
Average all		4597.2	1263.5	702.6							
Average without 1st up curve		4272.6	1217.6	682.6		Average without 1st up curve		#DIV/0!	#DIV/0!	#DIV/0!	

Average Viscosity Data

Viscosity in cP	**AZ-102 Mixing and Aging**	25 C	25 C	25 C	25 C
File Name	Material	33 1/s	150 1/s	298 1/s	Other?
061900A up (0-300 1/s)	AZ-102 1hr	275.9	89.6	53.0	
061900A down (300-0 1/s)	AZ-102 1hr	247.1	81.2	50.9	
061900B up (0-300 1/s)	AZ-102 5 1hr - R	254.8	77.5	48.1	
061900B down (300-0 1/s)	AZ-102 1hr - R	232.3	72.7	46.1	
Average all		252.5	80.3	49.5	
Average without 1st up curve		244.7	77.2	48.3	
062000A up (0-300 1/s)	AZ-102 ~24 hr	143.9	45.5	29.11	
062000A down (300-0 1/s)	AZ-102 ~24 hr	113.2	43.3	26.3	
062000B up (0-300 1/s)	AZ-102 ~24 hr - R	119.8	40.1	26.9	
062000B down (300-0 1/s)	AZ-102 ~24 hr - R	116.9	37	26.1	
Average all		123.5	41.5	27.1	
Average without 1st up curve		116.6	40.1	26.4	
062600A up (0-300 1/s)	AZ-102 ~ 1 week	80.59	23.6	16.94	
062600A down (300-0 1/s)	AZ-102 ~ 1 week	47.8	19.7	14.66	
062600B up (0-300 1/s)	AZ-102 ~ 1 week - R	66.29	24.29	16.65	
062600B down (300-0 1/s)	AZ-102 ~ 1 week - R	51.96	18.7	14.81	
Average all		61.7	21.6	15.8	
Average without 1st up curve		55.4	20.9	15.4	
072800A up (0-300 1/s)	AZ-102 Aged ~ 1 week	457	120.3	64.7	
072800A down (300-0 1/s)	AZ-102 Aged ~ 1 week	232	86.44	62.8	
072800B up (0-300 1/s)	AZ-102 Aged ~ 1 week - R	274.1	94.7	57.9	
072800B down (300-0 1/s)	AZ-102 Aged ~ 1 week - R	227.2	84.3	58.5	
072800C up (0-300 1/s)	AZ-102 Aged ~ 1 week - R	246.3	85.4	54.4	
072800C down (300-0 1/s)	AZ-102 Aged ~ 1 week - R	222.8	81.8	54	
Average all		276.6	92.2	58.7	
Average without 1st up curve		240.5	86.5	57.5	

APPENDIX C

Appendix C: Test Instruction

PNNL Test Instruction	Document No.: BNFL-TI-29953-082 Rev. No.: 0
Title: Preparation of simulated Sr/TRU Removal Solids for Rheology Testing	
Work Location: PSL, 1425	**Page** 1 of 9
Author: Richard T. Hallen	**Effective Date:** 3/1/00 **Supercedes Date:** New
Use Category Identification: Information	
Identified Hazards: __Radiological _x_ Hazardous Materials __ Physical Hazards __ Hazardous Environment __ Other:	**Required Reviewers:** _X_Technical Reviewer

Are One-Time Modifications Allowed to this Test Instruction? _X_Yes __ No

NOTE: If Yes, then modifications are not anticipated to impact safety. For documentation requirements of a modification see SBMS or the controlling Project QA Plan as appropriate.

On-The Job Training Required? ____ Yes or __X__No

FOR REVISIONS:
Is retraining to this procedure required? ____Yes __X__No

Does the OJT package associated with this procedure require revision to reflect procedure changes?
____Yes _____No ___X___N/A

Approval	Signature	Date
Author	Richard T. Hallen RH	3/2/00
Technical Reviewer	D E Kurath	3/2/00

Controlled Document

5.0 Test Instructions for Solids Preparation from Permanganate Treatment

The laboratory record book (LRB) shall be used to record other testing information as required by this procedure and all test conditions not stated by this procedure.

5.1 Pre-start for Scoping Studies

5.1.1 Inventory materials, equipment, supplies, and reagents to ensure all required items are available.

5.1.2 Do the following and initial and date when each item is completed.

DH ______ Review the work instructions in this test instruction before beginning. Any questions should be addressed to Dean Kurath or Rich Hallen. And questions need to be answered before proceeding.

Log Book # BNW 13733

5.1.3 Obtain the following information:

M&TE List:

DH Balance 1:

Calib ID 380-06-01-006 Calib Exp Date 2/01

Location PSL 1425

DH ~~Balance 2:~~ 380-06-01-013

~~Calib ID ______~~ Calib Exp Date 2/01

~~Location ______~~ PSL 1425

~~Thermocouple:~~

~~Calib ID ______~~ ~~Calib Exp Date ______~~

~~Location ______~~ ~~Thermocouple type ______~~

~~Digital~~ Thermometer:

Calib ID ______ Calib Exp Date ______

Location ______

ASTM 90C 23.8°C = 23 °C ERTCO R 2241

Kessler 811-066

5.2 Permanganate Treatment of AN-107

5.2.1 Turn on stirrer.

5.2.2 While continuously stirring the waste add the metal salts to the simulant. The may not dissolve or may dissolve and precipitate but this is expected.

$Fe(NO3)3$ Target amount	56.81 grams	Actual amount	56.8398 grams	dissolved in 205 H_2O
$Ca(NO3)2$ Target amount	~~7.10~~ 2.86 grams	Actual amount	2.9032 grams	
$Cr(NO3)3$ Target amount	~~2.84~~ 1.85 grams	Actual amount	1.8319 grams	
$Pb(NO3)2$ Target amount	~~1.24~~ 0.77 grams	Actual amount	0.7844 grams	
$MnCl_2 \cdot 4H_2O$	7.10		7.1019 grams	

5.2.3 Collect a single 10 mL sample for analysis via ICP.

duplicate SM-01 + SM-02 collected 10:10 AM

5.2.4 While continuously stirring the waste sample, personnel are to slowly add 599.97mL of 1.0 M $Sr(NO_3)_2$ solution. (based upon BNF-003-98-0023, add 1M Sr at a rate of 2 to 20 mL/min/L of waste, target 10 mL/min/L of waste or 70 mL/min) 10:20 addition started

Tare ~~bottle~~ beaker of 1M Sr 1347.52 g 10:30 completed
Tare Empty ~~bottle~~ 407.00 g
Weight Added ________

5.2.5 While continuously stirring the waste sample, personnel are to slowly add 399.98 mL of 1 M $NaMnO_4$ solution. (based upon BNF-003-98-0023, add 1 M NaMnO4 at a rate of 2 to 20 mL/min/L of waste, target 10mL/min/L of waste or 70 mL/min) 10:35 addition started

Tare ~~bottle~~ beaker of 1M NaMnO4 976.22 g
Tare Empty ~~bottle~~ 415.20 g 10:45 completed
Weight Added ________

Stir mixture for 30 minutes

5.2.6 Heat the waste mixture at $50 \pm 5^{\circ}C$ with stirring for 4 hours after completing the addition of the NaMnO4.

11:17 - both hot plates turned on
- third hot plate added

5.2.7 Turn off the stirrer and allow the waste to cool to $25 \pm 5^{\circ}C$.

11:43 t = 38°C
glass wool blanket added to keep in more heat
12:10 - t = 45°C
12:32 t = 50°C hot plates turned down to Low.
13:58 t = 49°C

5.3 Solids Removal with the Deadend Filter Unit

~~5.3.1 Tare the filter assembly/filter media.~~

~~Tare of the filter assembly ________ grams~~

Not done

5.3.3 Assemble a large filtration unit and filter all of treated waste in the treated simulant. The solids should have settled on cooling/setting. Decant all of the supernate from the treated waste to the filter. The supernate should filter faster without all of the solids/cake on the filter unit.

at 3:10/15:10 ~100 mL slurry sampled

14:25 t = 49°C
15:02 t = 48°C
15:39 t = 46°C
16:00 heat-hot plates turned off
16:10 44°C
16:45 stirrer removed

Collect duplicate samples of the damp sludge for analysis by ICP and IC. A single simulant sample should be collected after the shim chemicals are added for analysis by ICP.

SM-05 dried solids from Jar #1

SM-06 dried solids from Jar #2

final supernatant density

25 mL = 30.9474 grams

d = 1.2379 g/mL

wt % solids	tare	dry weight			wet solids	Dry Sol
Jar #1	3~~6~~5.4644	36.3186	36.3095	36.3129	3.1817	0.8469
Jar #2	18.2687	19.2481	19.2363	19.2387	3.7262	0.9688

Jar #1 26.61 wt % solids

Jar #2 26.00 wt % solids

- NOT SUPRISING THAT #1 is a little wetter, they were placed in Jar & Sealed sooner. Jar #2 was less solids & after some period of time — probably 10 minutes between sealing of the two bottles/Jars.

wrong direction? would have expected this

RHalin
3/6/00

Formula	grams	ug/g	mg/L	mmol/L	g/L
$Ca(NO_3)_2 \cdot 4H_2O$	236.15	50	63.55	1.585579	0.374434
$Cr(NO_3)_3 \cdot 9H_2O$	400.15	25	31.775	0.611105	0.244534
$Fe(NO_3)_3 \cdot 9H_2O$	404	800	1016.8	18.20689	7.355582
$Pb(NO_3)_2$	331.2	50	63.55	0.306708	0.101582
$MnCl_2 \cdot 4H_2O$	197.9	200	254.2	4.627034	0.91569

Treatment of AN-107 simulant
per liter of waste

1M Sr	0.0857 Liter or	0.17142 moles
1M MnO_4	0.0571 Liter or	0.11428 moles

Balance number: ________________.
Calibration date: ________________

280.03 grams

Actual

Makeup $Sr(NO_3)_2$ solution
use $Sr(NO_3)_2$
211.63 grams/mole (FW) (lot #)
Tare ~~Bottle/flask~~ ________________ grams
add 36.278 grams $Sr(NO_3)_2$ ~~per liter of simulant~~ 280.39 grams
~~for each liter treated~~ add 85.71 gram H_2O per liter treated ________ grams
~~actual weight of~~ $Sr(NO_3)_2$ added ________ grams
661.6 g actual weight of water added 661.65 grams

~~Label bottle with ID~~,

Makeup $NaMnO_4$ solution
use
$NaMnO_4 \cdot H_2O$
159.94 grams/mole (FW) (lot #)

141.09 g

actual

Tare ~~Bottle/flask~~ ________________ grams
add 18.278 grams $NaMnO_4$ ~~per liter of simulant~~ 141.11 grams
~~for each liter treated~~ add 57.14 gram H_2O per liter treated ________ grams
~~actual weight~~ of $KMnO_4$ added ________ grams
actual weight of water added 421.30 grams

Date prepared:
Prepared by:
Work Package Number:

441.07 g

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